Effect of Size Level and Fiber Coarseness on Ink-Jet Printability of Size Press Coated Paper

Falguni Mehta
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Falguni Mehta
EFFECT OF SIZE LEVEL AND FIBER COARSENESS ON INK-JET PRINTABILITY OF SIZE PRESS COATED PAPER

Falguni Mehta, M.S.
Western Michigan University, 2000

In recent years, matte coated paper has enjoyed wide spread usage for ink-jet applications. The coating performance is strongly influenced by base sheet properties including fiber coarseness and base sheet absorbency. Base sheet absorbency, which is evaluated in terms of internal sizing, is a crucial property of ink-jet paper.

The objective of this research was to evaluate the influence of size level and fiber coarseness on ink-jet print qualities. A secondary objective was to examine the size reversion phenomena and to quantify the effect of size level, pigmented coating, and fiber coarseness on size reversion.

The results showed that the print density was higher for the sheets made from coarser species. It increased with size level, and decreased with coating level. Delta gloss increased with coating level. Sheets made from coarser species had poorer grid roundness values. Size reversion was observed for all the samples with time. Size reversion was not significantly influenced by coarseness, size level, and coating level.
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CHAPTER I

INTRODUCTION

After being developed in the early 1980s, ink-jet printing technology is now enjoying wide usage in the fields of business application and computer graphics. Ink-jet printing is a non-impact printing process, and produces an image by transferring individual drops of ink from an orifice through a small air gap to a variety of printing surfaces, including corrugated board, plastics, fabrics, and paper. Dynamics of ink interaction with the paper surface during an ink-jet printing process depend in a complex way on the surface chemistry and physical properties of the sheet (1).

The expansion of ink-jet into additional printing markets and relegation of most other digital color printing technologies to niche markets may begin to challenge electrophotography in many high-end applications. Some of the properties that an ink-jet recording media should have are given below (2):

1. The base sheet should have uniform formation to achieve uniform coating and high print quality.

2. The coated film should have small pore size compared to the ink drops to avoid wicking or streaking.
3. The paper surface should maximize color density and contrast, and minimize show through.

4. The paper should have excellent smoothness, gloss, waterfastness, lightfastness, and rub resistance.

5. The coated film must be able to absorb ink drops quickly to avoid image smearing and multiple droplet splatter.

6. The coating surface should have a high porosity and a high surface area that wets virtually instantaneously with water. It should be of sufficient thickness to absorb successive droplets in multicolor printing, at the highest delivery rates without becoming saturated with ink.

7. The coating should not dust or lint.

8. The coating should reduce the consumption of ink for desired print quality.

9. The coating should have a nearly neutral or alkaline pH.

10. The base sheet should be adequately sized to prevent the ink penetration through it.

Examining these requirements, it is apparent that a hydrophilic and a uniform coating, on an adequate hydrophobic and uniformly formed base sheet, is a critical requirement for any ink-jet media. Therefore, the base sheet plays a significant role in determining coating formulation parameters required to achieve the required property balance (3). Besides the base sheet
formation, two additional requirements needed to produce high quality ink-jet papers are: (1) a highly hydrophilic coating, containing suitable pigment and binder formulations; (2) a highly hydrophobic base sheet (internal sizing), containing suitable sizing agent with compatible retention aids for retaining the sizing agent.

Advances in ink-jet printing technology have resulted in a new generation of ink-jet printers that can print faster on wider paper widths and produce four color images close to photographic quality. Ink-jet papers must be tailored to these changes through quick ink absorption, minimizing ink bleed and ink wicking, while retaining favorable ink optical density values. To meet these demands, many papermakers are turning to pigmented size press coating (4). For pigmented coatings, different pigments are available. Among them, precipitated calcium carbonate would be a preferred pigment, as it gives good brightness, opacity, ink receptivity, smoothness, and a highly finished coating at an economical rate. For binding the pigment particles, PVOH is commonly used due to its high binding strength.

Sizing of the base sheet plays an important role in the physical dynamics related to the print quality. Most ink-jet inks for home and office are partially water based. So, alkyl ketene dimer (AKD), the most effective and hydrophobic internal sizing agent, is a good choice for ink-jet applications to prevent water penetration further into the base sheet (5).
CHAPTER II

LITERATURE REVIEW

Ink-jet Printing

Ink-jet printing is the first digital printing technology that has achieved an acceptable level of color quality at an affordable price, for the majority of end users (6). Ink-jet printing is a radical departure from conventional printing and reprographic technologies. It has become widespread, as it produces little noise and makes high-speed printing possible. It also provides the additional benefits of four color printing at a fraction of the cost of color laser printing. Basically, it relies on computer input to form a dot matrix image by ejection of uniformly shaped droplets of ink or aqueous dye solutions (7).

Principle of Ink-jet Image Production

In an ink-jet printing system, images are produced on the recording surface by an electronically controlled flow of high velocity, fine fluid ink droplets or tiny solid ink streams. The ink drops are controlled by digital signals to produce images on the ink-jet paper surface and this differs from
conventional printing methods that use pigmented inks and direct transfer the images through contact with an inked image carrier (1).

**Types of Ink-jet Printer**

Following are the two major types of ink-jet printers that are currently available in the market (8).

1. **Drop on demand system:** In this system, a drop is produced only when it is required to form the image. It has inherent low speed characteristics and is used in desktop and graphic arts printing.

2. **Continuous ink-jet system:** In this system, each ink orifice sprays ink drops continuously towards the printing surface. The drops not needed are deflected to prevent them hitting on the printing surface, and accumulated in the ink recycle unit for reuse. It has inherent high-speed characteristics and is used in industrial and graphic arts printing.

**Types of Ink-jet Media**

Following are the three major types of ink-jet media available in the market (1).

1. **Plain or uncoated paper:** This is an inexpensive grade ($ 0.01/sheet) with poor color gamut, limited coverage, high bleed, strike through, cockle/curl, and is used for low quality output.
2. Glossy paper: This is the most expensive grade ($0.60 – 0.80/sheet) with nonporous structure, slow drying rate, poor water fastness, and is used in very high quality color images and for photographic quality images.

3. Matte coated paper: This is of an intermediate cost ($0.05 – 0.20/sheet) with good color gamut, fast drying time, excellent resolution, 100% coverage, and is used for overall best performance.

The difference in the ink absorption phenomenon for the different grades is shown in Figure 1. For plain paper, the ink absorption is non-uniform causing more bleeding than other grades. For matte grade, the ink drop stays in the coating layer producing a good color gamut. For photo grade, the ink stays in the polymeric layer increasing the drying time but giving best print quality (9):

![Figure 1. Ink Absorption Phenomenon for Different Ink-jet Grades.](image)

Matte or low gloss (<33) paper comprise about 25% [North America] and 50% [Europe] of coated paper production. Matte grade represents a
significantly growing segment of the coated paper market as it provides less glare and easy readability. Ideally, a matte pigmented coating system should produce a high brightness coating with high delta gloss and low print abrasion. The most important criteria for a matte grade paper are good printability with high delta gloss (3).

**Ink-jet Inks**

Ink-jet printing has seen explosive growth in the past decade and the range of printer and ink types continues to multiply. Inks must be formulated to meet the end use demands while being compatible with broad variety of delivery devises. Ink performance is generally dependent on choice of printing substrate. Different ink and different substrate properties are necessary to produce a good quality image (1). The physical properties needed for ink-jet inks are: (1) ink composition that does not cause nozzle clogging; (2) ink must not have any chemical effect on nozzles; (3) ink must be water resistant and weather proof; (4) must produce high quality printing without causing bleeding. The ink dot control is a principle criteria for ink-jet printing that includes: (a) presence of all dots, (b) circular dots, (c) proper size of all dots, and (d) same size and appearance of all dots on different media.
Areas of Application

Currently, ink-jet printing is used in many different areas such as those shown below:

1. Home/Office: In recent years, desktop ink-jet printers have made rapid advances in the home and offices due to convergence of hardware and software technologies.

2. Industrial: Several types of printing technology (e.g. continuous and impulse) are used industrially, each with a rigorous set of requirements regarding ink formulation driven by critical application factors such as speed, barcode readability and durability.

3. Wide format: Ink-jet printing is replacing traditional printing techniques to create large images in such application as posters, signage and billboard printing.

4. Other applications: High speed postal addressing, wire/cable marking, personal check printing, barcode marking, label printing, and carpet printing.
Fiber Coarseness

Paper plays an important role in determining image quality in ink-jet printing. Performance of the paper depends on many factors such as furnish (e.g. wood fiber length, coarseness, cell wall thickness, stiffness), formation, additives etc. So, the dimensions of the raw wood fibers, used in papermaking are crucial in the development of paper properties, including strength, optical and surface properties. Among all these, fiber coarseness is one of the important fundamental properties. Just as the fineness of wool or cotton or silk has a profound effect on the fabric qualities, fineness or slenderness of pulp fiber has on paper, and so on its printing properties. The fiber coarseness affects not only the strength of paper, but also its air and liquid permeability, surface roughness, and hence, printing quality (10).

Coarseness of wood fiber is expressed in decigrex (dg.), the unit being one tenth of a grex, thus equal to milligrams per 100 meters of oven dry fiber. Coarser fibers have a dg. over 30 and very fine ones have a dg. less than 10. Coarseness seems to be most attributed to denser and longer fiber.

Figure 2 shows that long fibers are coarser than short ones. The increase in coarseness with length may be due to an increase in cell width,
cell wall thickness, or both. So the main morphological features i.e. length, width, and wall thickness, are all interrelated (11).

![Graph showing Fiber Coarseness vs. Fiber Length](image)

Figure 2. Fiber Coarseness vs. Fiber Length.

The coarseness of a fiber largely depends on the difference between the area of its entire cross section to that of its lumen, or central canal. It depends on the average diameter of a fiber cross-section and the thickness and density of its walls (10). Fibers with thick cell walls are more rigid, which limits their response to consolidation forces during the papermaking process. This results in a more open structure, with higher light scattering coefficient, bulk, and porosity. Cell wall thickness usually tracks fiber length and is one of the most important properties in determining fiber coarseness. Generally, thick-walled fibers of small diameter do not collapse in pulping; they are stiff, and retain their rounded shape during sheet formation. They resist beating and do not bind readily, but may improve tearing strength in the resultant sheet.
Such bulky sheets have low density and produce surfaces, which are rougher and more opaque, as well as, lower in burst and tensile strengths. While, wide, thin-walled fibers show the contrast behavior and collapse in the conversion steps, forming compact, dense sheets that are low in opacity, uniform in formation, and smooth (11). In chemical pulping, coarseness decreases during cooking as, organic matter is removed from the fiber wall and flexibility increases (12). Changes in fiber coarseness influence all pulp properties especially formation. The poor base stock formation influences coated paper quality by contributing to a non-uniform coat weight distribution. Better formation produces more uniform pore structure of the base stock and the coating layer. Improved formation reduces the bulk and roughness, and increases gloss and the light scattering coefficient of the coated paper (13).

**Internal Sizing**

Internal sizing plays a pivotal role in the physical dynamics in ink-jet print quality and determines to a large extent print sharpness and color vividness. Thus, the performance of ink-jet printers is greatly influenced by internal sizing agents, and the quality of ink-jet paper can be evaluated as a function of type and amount of internal size (5).
Sizing chemicals are specifically added to the base sheet to regulate or retard liquid penetration. They can also affect the optical, strength, and surface properties of the ink-jet substrate. Ink-jet printing is particularly susceptible to the effects of drying due to the mechanism of ink placement and spreading on the sheet. The size level of the sheet is monitored by measuring the contact angle of fiber against water and also by macroscopic sheet sizing test.

Internal sizing agents provide a reduced rate of liquid penetration by retarding the rate of flow through the inter-fiber capillaries. Figure 3 shows the ideal behavior that is described by the Lucas Washburn Equation (15). If the contact angle is 90° or greater, there is no penetration.

\[
\frac{dl}{dt} = \frac{\gamma \times r \times \cos \theta}{4 \times \eta \times L}
\]

**Legend.** \( dl/dt \) = rate of liquid penetration, \( \gamma \) = surface tension of fluid, \( r \) = radius of capillary, \( \cos \theta \) = degree of wetting of fiber surface, \( \eta \) = viscosity of fluid, \( L \) = distance penetrated

Figure 3. Lucas Washburn Equation.

Alkyl Ketene Dimer (AKD), which is a hydrophobic and low surface energy sizing agent, increases the contact angle of the liquid at the fiber
surface, by attaching itself to the hydrophilic end of fiber and is used to meet the sizing requirements in most alkaline systems. It is used where hard sizing against aggressive penetration is desired, as well as, in a wide range of slack and moderately size paper, or when sizing is required for controlling pickup of surface treatment (15).

![Figure 4. Mechanism of Sizing With Alkyl Ketene Dimer.](image)

For successful completion of this reaction (Figure 4), the AKD is first emulsified to a certain degree to get the stable form of AKD. These emulsified particles of AKD must be retained with the help of cationic material (Retention Aid) on the fiber surface. Once the retention is obtained, the fibers are consolidated simultaneously to the web. Buildup of heat in the dryer section begins melting and spreading of the particles over the fiber surface. The AKD must spread from a particle 0.2 – 2.0 µ in diameter to a monomolecular layer, covering the fiber surface. Theoretically, 0.5 pounds per ton would provide a monomolecular layer over most pulps. In reality, an
excess must be used to compensate for a non-uniform distribution, retention losses, unreacted size and size lost due to competing reactions (hydrolysis of AKD to form ketone that is detrimental for sizing) (15). In comparison of AKD and ASA (Alkynyl Succinic Anhydride), AKD generates better optical density for ink-jet printing at the same sizing level with reduced color to color bleed, as the rate of ink penetration is slower than ASA sized sheet (5).

**Pigmented Coating**

The customers of ink-jet paper demand high fidelity and vibrancy from color printing but usually hesitate to purchase expensive, high quality coated papers. As a result, manufacturers are challenged to satisfy them with inexpensive pigmented products that perform well on different printers (16). Paper structure and surface chemistry requirements for good print quality are so unconventional that new paper grades, specially designed for ink-jet printing, are continuously being formulated (7). Pigmented surface coatings applied at the size press during the paper making process can be used to modify the chemistry of the paper surface, and improve the performance of the paper in different printing environments, especially in the case of non impact printing. Coating a paper improves its surface finish, optical characteristics, and printability. The development of macroporosity of the coating is a function of particle size distribution, pigment/binder ratio,
additives, and the chemical/physical nature of the binder. The coating structure can describe the final properties of paper. This general term encompasses parameters like coating thickness, porosity, pore size distribution, and the distribution of coating ingredients at the surface and within the paper. Control of this structure is the most important issue of the coating process (13). The structure of the coating layer is influenced by many factors including: (a) base stock properties, (b) coating color composition, (c) color and base sheet interactions, (d) application and metering method, and (e) drying and calendering of the coated web.

The reasons for pigmented ink-jet coating are explained below:

1. Improves appearance before printing by improving brightness, gloss, smoothness, color, and uniformity of appearance.

2. Improves appearance after printing by improving gloss, ink hold out, ink transfer, print details, controlled mottle, show through, and strike through.

3. Improves mechanical strength of surface (wet/dry), glueability, and reduces ink consumption.

Calcium carbonate, sometimes referred to as chalk, has been used as a pigment since ancient times to impart brightness, opacity, smoothness, ink receptivity, and finish to the coating. It is used alone or in combination with other pigments, to impart the desired coating characteristic. There are two
general types of calcium carbonate: natural and precipitated. Natural is very pure limestone, which is ground to a very fine powder, while precipitated calcium carbonate is a prime or byproduct of the alkaline pulping industries and others. It is a product of excellent brightness (96% – 98%), fine particle size (0.35 – 2.2 µm) and specific gravity of 2.7. It adds openness, porosity and high finish to the coated layer. At any given particle size it will be lower in gloss and more ink absorbent than clay, and thus is a good pigment for matte coated ink-jet papers (3).

Roll performed by Binders in pigmented coating:

1. In coating color before coating: (a) compatible with pigments, (b) thermally, mechanically, and chemically stable, and (c) control of flow behavior.

2. During coating: (a) response to high shears, (b) leveling and migration during drying, and (c) water retention and fast drying rate.

3. In final coated paper: (a) binding - pigment to pigment, pigment to substrate, and within substrate, (b) resistant - dusting and creaking at folds, abrasion, water, and blister, and (c) others - pick strength, flexibility, glossing response, dimensional stability, ink receptivity, ink hold out, glueability.

Polyvinyl alcohol (PVOH) is a preferred polymer binder for ink-jet coating system. Coatings based on high surface area or high void volume pigments in combination with PVOH binder are evolving as the preferred
coating for high performance ink-jet printing. For pigmented ink-jet coatings, the performance characteristics of PVOH relate to the polymer's hydrophilic nature, its inherent high binding strength, and its nonionic character that makes it compatible with other additives in the formulation. Laboratory coating methods have shown that fully hydrolyzed grades are generally best for lightly pigmented size press applications, where as, partially hydrolyzed grades are better suited for highly pigmented matte coated grades. Depending on the molecular weight, PVOH can be three to four times as strong as starch. Its use permits significantly reduced binder levels (Figure 5) in size press formulations and the maximum utilization of the pigment's high absorption capabilities without dusting or linting problems. Also PVOH is superior to typical latex binders that produce ink-jet images that are mottled, low in ink density, and provide poor ink bleed control (4)

![Diagram](Figure 5. PVOH vs. Starch.)
Interaction in Fiber Coarseness, Internal Sizing, and Pigmented Coating

Paper is coated in order to provide a smooth surface, which gives a detailed, uniform, defect free images when printed. The coated surface has a much better working relationship with the printing process than the surface of uncoated paper, especially when paper contains large amounts of coarse fibers. For pigmented size press coatings, optimum print quality is obtained by a judicious balance between internal sizing, and a pigmented size press treatment. And all the action of sizing and coating will be greatly affected by the base sheet properties, especially by fiber properties.

Wood fiber coarseness, measured as a function of fiber length, cell wall thickness, fiber cross sectional area, and fiber stiffness, has tremendous effect on paper strength, air and liquid permeability, surface roughness (poor formation), and printing quality. To suppress the effects imparted by fiber coarseness, coating can be used (13).

Figure 6 shows the combined effect of coarseness and coating on the PPS roughness before and after coating.
In case of applying color to the substrate, the volume of ink required is much higher for ink-jet printing applications compared to other commercial printing operations. This generates some significant application problems for the ink-jet process, since the inks contain a large amount of water (65-90% by weight). Plain, sized and surface treated papers have been developed to reduce the problems associated with the high volume of ink, used in ink-jet printing. As shown in Figure 7, plain papers absorb ink very quickly, reducing the dot circularity and increasing dot size. Sizing the paper, reduces the ink absorption into the paper resulting in a delay in the colorant drying and generating a little color to color bleed. Papers coated with a hydrophilic coating that contains highly porous and interactive pigments, and water soluble polymers produce the best printing quality with the least amount of defects (2).
So, for non-impact ink-jet printing, the coating must be hydrophilic to absorb ink (water) quickly and thick enough to prevent further ink penetration into base sheet. If ink penetrates into the base sheet, it will result in a distorted print image. This can be prevented by means of a hydrophobic base stock. Thus the interaction of the internal sizing with hydrophilic coating is of critical importance in achieving adequate ink-jet quality.

Based on the above analysis, all the three variables are interrelated to give optimum ink-jet print quality or sharp images.

**Size Reversion**

Size reversion is the natural loss in internal sizing with time, under ambient conditions. Loss of sizing, at the size press or as a result of other chemical or physical treatments, is a separate phenomenon from above mentioned size reversion. Size reversion is a phenomenon, both aggravating
and expensive, and currently suffered by many producers of alkaline papers. Sizing agents are added to the paper at a level adequate to maintain some minimal resistance to liquid penetration, required for subsequent surface treatments or end use requirements. When the extent of reversion is large, the amount of sizing agent required becomes excessive. Not only are sizing agents expensive, but in the case of AKD, excessive size can lead to problems with surface slippage. The cause of size reversion is as elusive as, sizing itself. Without clear understanding of the reversion mechanisms, it is impossible to devise a rational strategy for suppressing it. The loss of sizing with time, in AKD sized papers, is due to an environmentally induced molecular reorganization of the AKD-modified fiber surfaces (the energetic discomfort of AKD's long chain alkyl subsistent in a moist environment, driving them to reorganize in such a way as decrease their free energy) (17).

**Mechanism**

The hydrophobic AKD molecules form a high-energy interface with atmospheres of typical levels of humidity. The alkyl chains of the AKD will reorganize, in such a way as to minimize their surface energy. This results in increased exposure of fiber surfaces and diminished water repellency (16).
Cause

Figure 8 shows the migration of unbound and mobile AKD molecule exposes a region of the fiber surface.

Figure 8. Size Reversion Phenomenon.

Figure 8 illustrates a conceptual AKD-sized fiber surface. Circles depict the reactive polar end. For sake of simplicity, single rather than double alkyl tails are shown for each AKD molecules. Five of the AKD molecules have reacted (bounded) with the fiber surface and one is free (unbounded). In the dry environment of the dryer a uniform coverage of the fiber surfaces with the hydrophobic AKD is energetically favorable. The coverage of the same fiber surface after the unbound AKD has been removed with chloroform environment is illustrated in the second figure. An area of the surface is now left exposed for easy penetration by water, and sizing is reduced. When the same surface is exposed to 50% RH, as with the original droplets of the AKD emulsion particles on the fiber surface, the AKD molecules will try to minimize the surface area that they expose to this moist environment. They will do this by reorganizing themselves, to the extent that
they are able, given that a portion of them are now permanently anchored at one end. So, this figure illustrates the migration of unbound and more mobile AKD molecule that exposes a region of the fiber surface (16).

**Solution**

The solutions to reduce or prevent the size reversion are explained below:

1. Reduce the mobility of AKD molecules, by maximizing the proportion of these covalently bonded molecules to the fiber surface.

2. Increase the uniformity of AKD coverage on fiber surface, to decrease the exposure of fiber after alkyl chains have reorganized.

3. Reduce the amount of unbounded AKD molecules by not only retaining the size in the sheet, but by fixing it selectively to the fibers.

Cationic starch is particularly effective retention aid in optimizing the performance of AKD size both by fixing AKD to fiber and doing so in a uniform manner (17). Figure 9 shows the effect of starch dose on AKD sizing response (here the sheet were made using 15% PCC and 2.5 lb./ton AKD).

Cationic starch increases the dispersion stability of cationic AKD emulsion particles and deposits them on anionic fiber surfaces as discrete entities. High cationic starches are most effective at suppressing size reversion for this reason.
So, optimizing AKD sizing requires maximizing initial sizing efficiency and minimizing the extent of reversion. Strategies for optimizing the amount of AKD retained, and optimizing the uniformity of its distribution will accomplish both causes.
CHAPTER III

STATEMENT OF PROBLEMS AND OBJECTIVES

Although it is a fact that the combination of hydrophilic coating and hydrophobic sizing is a crucial requirement for the non-impact printing media, it is necessary to know the effect of coarseness, size level, and coating level on the paper, as well as, print properties of ink-jet substrate. Also, it is important to emphasize the effect of these control variables on size reversion.

To determine the effect of fiber coarseness individual base sheets were produced using southern and northern softwood species. In this research, PCC and PVOH were used for the size press pigmented coating formulation at three different percentages of coating solids. AKD was used at four different levels in combination with cationic starch for producing sized base sheets with different amounts of alkyl ketene dimer.

The primary objective of this thesis was to determine the influence of size level and fiber coarseness on the print properties of ink-jet papers coated with the above formulations on a flooded nip untied size press. The secondary goal was to examine the size reversion phenomenon for all the substrates produced for this research.
CHAPTER IV

EXPERIMENTAL DESIGN AND METHODOLOGY

The experimental design used during this study was designed to investigate the influence of coarseness and base sheet sizing on the ink-jet printability. The research was divided into five phases.

Phase I: Base Sheet Production

Phase I involves the production of the base sheet. The base paper was made on Western Michigan University’s pilot plant paper machine in the Paper and Printing Science and Engineering Department. The base paper consisted of a 60/40 Hardwood to Softwood mixture. This trial was done separately for Northern and Southern softwood species. The stock was refined in a double disk refiner at 60 gpm and 45 KW for 25 minutes to a freeness of 350±25CSF. Alkaline Ketene Dimer (AKD) was used as a sizing agent. AKD was added at four different levels (2, 3, 4 and 6 lb./ton). Cationic starch was added to improve the retention of AKD. Precipitated calcium carbonate (Albacar HO) was used as filler. PCC (Albagloss SF) was used as a coating pigment in combination with PVOH (Airvol 107) binder. The
coating formulae were applied with the paper machine’s flooded nip untied sizepress (vertical) at three different levels of solids (18%, 22%, and 25%). The paper was calendered on-line through three nips at 40 psi. The trial plan for the base sheet production is given in the appendix I.

Phase II: Measurement of Paper Properties

In this phase, the properties of the coated and uncoated samples were measured. Table 1 shows the measured paper properties and the equipment used for testing those particular properties.

Table 1

<table>
<thead>
<tr>
<th>Test</th>
<th>Equipment</th>
</tr>
</thead>
<tbody>
<tr>
<td>Absorbency</td>
<td>Hercules Sizing Tester</td>
</tr>
<tr>
<td></td>
<td>First 10 Å Dynamic Contact Angle</td>
</tr>
<tr>
<td>Coarseness</td>
<td>Kajaani Analyzer</td>
</tr>
<tr>
<td>PPS roughness</td>
<td>Parker Print Surf ME 90</td>
</tr>
<tr>
<td>PPS porosity</td>
<td>Parker Print Surf ME 90</td>
</tr>
<tr>
<td>Brightness</td>
<td>Technidyne Brightimeter</td>
</tr>
<tr>
<td>Opacity</td>
<td>Opacimeter</td>
</tr>
</tbody>
</table>
Phase III: Ink-jet Printing

This phase covered the printing of the base sheets with Epson Stylus Pro ink-jet printer.

Phase IV: Print Quality Evaluation and Data Analysis

In this phase, printed papers were evaluated for ink-jet print quality by measuring different optical and surface properties of the printed image. The print properties and the equipment for measuring the properties are shown in the Table 2.

Table 2
Print Properties and Equipment

<table>
<thead>
<tr>
<th>Test</th>
<th>Equipment</th>
</tr>
</thead>
<tbody>
<tr>
<td>Image Analysis</td>
<td>Image Pro Plus Analyzer.</td>
</tr>
<tr>
<td>Delta Gloss</td>
<td>BYK – Gardner GmbH micro-glossmeter 60°</td>
</tr>
<tr>
<td>Print Density</td>
<td>X-Rite 418 Reflection Densitometer</td>
</tr>
</tbody>
</table>

Data Analysis

The result from this study was analyzed using SAS, a statistical software package. This analysis identified the significant primary effects and
significant interactions. The control variables in this study were fiber coarseness, AKD application level, and coating solids, while the principal response variables were those described above.

Phase V: Thesis Write-up

This phase covered the writing of the thesis summarizing all above phases.

Figure 10 shows the schematic diagram of various experimentation phases.

Figure 10. Schematic Representation of Various Phases of Research.
CHAPTER V

RESULTS AND DISCUSSION

For the research two separate base sheets were produced using southern and northern softwood species with different levels of size and coating. Then finally, determine their ink-jet printability to evaluate the influence of sizing, coating, and coarseness on the print properties. So, in this thesis, there are three control variables to examine: (1) size level, (2) coating levels, and (3) coarseness. The effect of these variables on paper and print properties were determined. So, all the paper and print properties are the response variables for this research.

Experimental Data Analysis

Data analysis was performed using SAS, a statistical software package, to analyze the collected data. In performing the data analysis, the General Linear Model (GLM) procedure was used. GLM handles missing data points and produces ANOVA tables. To observe the differences between means, for effects determined, to be significant Duncan's multiple range test was used. This test compares the mean of the highest value to the second highest value so on to the lowest value. In this way all treatment means for an effect were
compared to determine if any statistically significance exists between one treatment and another in a given effect.

In this experiment no replicates were performed. So, the data set with all measurements was used to obtain a good estimate of the experimental error. The analysis was performed on data sets, the means and the complete data set. This statistical analysis consisted of three phases is described below:

1. Phase I: This phase classified the dependent and independent variables and modeled the main effects to identify which variables are significant as determined by their P-values. The most significant variable is identified by having the highest F-value. The analysis is performed using one response variable at a time.

2. Phase II: This phase ran the model of main effects and interactions for each response variable. This phase identified if any main effect interactions contributed were significant. If a significant interaction was found, then the effect variable was paired with one significant main effect variable. The dominating main effect variable was then removed and the model was run with the other two main effect variables to evaluate their effects when one main effect is dominating.

3. Phase III: After removing the most significant main effect variable, the model was run with the other main effect variables, looking at their significance and comparing their means. If a main effect variable was
significant, Duncan’s comparison of means test was used to evaluate the significance of the main effect variable within the treatments performed for that effect. This test showed if any real significance exists between the means of the main effect evaluated.

Base Sheet Characterization

Two different base sheets were produced using southern and northern softwood species respectively at four different size levels using AKD as a sizing agent and cationic starch as a retention aid. The base sheets were size press coated at three different coating solids using PCC as the pigment and PVOH as a binder. Fiber coarseness for both the species was measured. The sheets were conditioned according to TAPPI standards. The paper properties including (a) absorbency, (b) Parker print surf (PPS) roughness, (c) Parker Print Surf (PPS) porosity, (d) opacity, and (e) brightness were measured. Hercules sizing test and dynamic contact angle test were performed to characterize the absorbency of the base sheet. The trial plan is shown in the Appendix A.

The coat weight obtained for each run is shown in the Table 6 of Appendix A. This shows that the size level played a significant role on the size press pick up or on the final coat weight. The coat weight decreased with increasing size level and increased with increasing coating solid.
Table 3 shows the coarseness of the two furnishes. The southern furnish has 21% higher coarseness value than the northern furnish. This shows that the addition of either southern or northern softwood fibers significantly affects the coarseness of the resulting furnish.

### Table 3

Fiber Coarseness

<table>
<thead>
<tr>
<th>Species</th>
<th>Coarseness in desigrex</th>
</tr>
</thead>
<tbody>
<tr>
<td>Southern species</td>
<td>14.50 d.g.</td>
</tr>
<tr>
<td>Northern species</td>
<td>11.46 d.g.</td>
</tr>
</tbody>
</table>

Table 4 shows the results of roughness, porosity, brightness, and opacity for the two base sheet produced during this trial. Size level did not significantly affect the optical properties. Roughness values are slightly higher for coarser species because coarser fibers are longer and may give poor and rough formation. PPS porosity values are higher for the coarser species because the coarser fibers give more open formation with more number of voids to pass the air. Size level did not make any change in porosity values because it affects the liquid absorptivity of the base sheet and may not deal with the air permeability.
Table 4

Base Sheet Properties

<table>
<thead>
<tr>
<th>Species</th>
<th>AKD lb/ton</th>
<th>Roughness (micron)</th>
<th>Porosity (ml/min)</th>
<th>Brightness (%)</th>
<th>Opacity (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Mean S.D</td>
<td>Mean S.D</td>
<td>Mean S.D</td>
<td>Mean S.D</td>
<td>Mean S.D</td>
</tr>
<tr>
<td>Northern</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>species</td>
<td>2</td>
<td>5.89 0.15</td>
<td>1310 54</td>
<td>90.94 0.14</td>
<td>88.98 0.66</td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>5.59 0.17</td>
<td>1276 46</td>
<td>91.10 0.14</td>
<td>91.18 0.43</td>
</tr>
<tr>
<td></td>
<td>4</td>
<td>5.84 0.10</td>
<td>1380 15</td>
<td>89.97 0.26</td>
<td>88.34 0.62</td>
</tr>
<tr>
<td></td>
<td>6</td>
<td>5.79 0.07</td>
<td>1172 48</td>
<td>89.57 0.16</td>
<td>90.38 0.13</td>
</tr>
<tr>
<td>Southern</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>species</td>
<td>2</td>
<td>6.06 0.08</td>
<td>1712 61</td>
<td>88.64 0.22</td>
<td>89.74 0.57</td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>6.06 0.12</td>
<td>1742 31</td>
<td>89.17 0.10</td>
<td>89.58 0.71</td>
</tr>
<tr>
<td></td>
<td>4</td>
<td>6.08 0.09</td>
<td>1787 97</td>
<td>89.91 0.15</td>
<td>87.46 0.62</td>
</tr>
<tr>
<td></td>
<td>6</td>
<td>5.95 0.11</td>
<td>1716 84</td>
<td>88.39 0.24</td>
<td>88.58 0.63</td>
</tr>
</tbody>
</table>

Table 5 shows the HST values of northern and southern base sheets. The HST values are higher for coarser species. This may be due to the lower surface area per unit weight of the coarser species. The 2 lb/ton sized sheets have very low HST values indicating that this level of AKD is insufficient for proper coverage of all the fibers and so, there are more open fibers in the sheet that increase the overall sheet absorbency. The absorptivity of the base
sheet continued to decrease with the level of sizing. And 6 lb/ton sized sheets have the highest HST values.

![Table 5](image)

**Table 5**

HST Results for Base Sheet

<table>
<thead>
<tr>
<th>Size Level (lb./ton)</th>
<th>HST of Southern Species (sec)</th>
<th>HST of Northern Species (sec)</th>
</tr>
</thead>
<tbody>
<tr>
<td>2</td>
<td>5.6</td>
<td>9.5</td>
</tr>
<tr>
<td>3</td>
<td>175.3</td>
<td>245.1</td>
</tr>
<tr>
<td>4</td>
<td>305.3</td>
<td>371.3</td>
</tr>
<tr>
<td>6</td>
<td>376.0</td>
<td>445.0</td>
</tr>
</tbody>
</table>

Effect of Coarseness, Size Level, and Coating Level on Paper Properties

**Absorptivity**

Because of its relationship to print quality, the absorptivity of the substrate is the primary property of interest. This was measured by Hercules Sizing Test and Dynamic Contact Angle test. The influence of size level, coating solids, and coarseness on absorptivity was determined by measuring HST and contact angle.
Hercules Sizing Test

This test uses a 2% formic acid solution of green color as a testing liquid. This liquid is poured onto the surface of the substrate. The time required for the solution to penetrate into the substrate is determined by measuring the reduction in the back reflection of light from the substrate. This test gives the time at which an 80% reduction in reflection occurs. So, it measures the resistance of the substrate to the penetration of a solution. An increase in the time shows more sizing and more resistant to liquid penetration.

Figure 11 and 12 show the effect of size level and coating levels on the HST of northern and southern species respectively. HST increased with increasing size level and decreased with increasing coating level. Coated sheets have higher absorbency than uncoated sheets. At the size press, there may be some penetration of the coating into the base sheet. This penetration may be responsible for the increased absorptivity of the sheet with increased coating solids. This penetration depends on the level of base sheet sizing. The penetration reduces with increasing size level. Coated 2 lb/ton sized base sheet was very absorptive. The sheets made from the coarser fiber have higher HST values.
Figure 11. Effect of Size Level on HST of Southern Species.

Figure 12. Effect of Size level on HST of Northern Species.
Dynamic Contact Angle

This test gives the contact angle values as a function of time for a particular substrate. It measures the rate of liquid penetration into the substrate with the time. Also, it can be used to follow the drop disappearing into the substrate. Distilled water of 7.5 pH was used as a testing liquid.

Figure 13 and 14 show the change in contact angle with time for both southern and northern species. Contact angle decreased with time for all the coated papers, as well as, with the coating level. This shows that on exposure to water, the water is gradually absorbed and that is absorbed faster at higher coating weights. This occurs because the porous coating layer is more absorptive than sized base sheet. Also, PCC in the coating is chemically interacting with the AKD and may reduce its effect. The contact angle of the uncoated sheet remained relatively constant after 60 seconds. The 2 lb/ton sized sheets have the lowest contact angle values and hence higher absorbency. This absorption is more profound with increasing coat weight. Overall, the contact angle increased with increasing size level and decreased with increasing coat weight. This is the same trend observed in the HST test. So, these data correlates with the data of HST test.
Figure 13. Effect of Size Level on Contact Angle of Southern Species.

Figure 14. Effect of Size Level on Contact Angle of Northern Species.
PPS Roughness

PPS Roughness was measured at a 500 lb. load and with a soft backing for all the samples.

Figures 15 and 16 show the influence of size level and coating solids on the PPS roughness. Roughness was not significantly influenced by size level. Sheets made from coarser wood fibers have higher roughness values. The coarse fibers are longer and may produce the rough formation. Roughness decreased with the application of coating. A uniform coating layer makes the surface smoother and hence roughness decreases.

Figure 15. Effect of Size Level on PPS Roughness of Southern Species.
Figure 16. Effect of Size Level on PPS Roughness of Northern Species.

**PPS Porosity**

PPS porosity, actually an air permeability, was measured at 1000 lb. load with smooth backing. This test measures the resistance to air penetration through the substrate.

Figure 17 and 18 show the effect of size level and coating solids on the porosity. Coated sheets have low porosity because the coating layer blocks in flow. As the coat weight increases the thickness of the coating layer increases, reducing the air penetration and hence permeability. Uncoated coarser species have higher porosity. The effect of coarseness on porosity diminished...
with the applied coating. Size level did not make any difference in the porosity values because sizing decreases the liquid absorptivity of the base sheet but does not interfere with the air penetration characteristic.

Figure 17. Effect of Size Level on PPS Porosity of Southern Species.

Figure 18. Effect of Size Level on PPS Porosity of Northern Species.
Opacity

Opacity was measured with a Technidyne opacimeter. This measures the hiding power of the substrate for incident light. As the refraction or scattering is higher, the substrate is more opaque. This refraction increases with particles of different refractive indices and more air voids.

Figure 19 and 20 show the influence of size level and coating solids on the opacity. Coated sheets have higher opacity and it increased with coating level. A thicker layer of coating increases the hiding power of the base sheet and the refraction of the light from the substrate surface. Because as the coat weight increases the number particles of different refractive indices increases, this increases the refraction. Size level and coarseness did not significantly influence opacity.

Figure 19. Effect of Size Level on Opacity of Southern Species.
Figure 20. Effect of Size Level on Opacity of Northern Species.

Brightness

TAPPI brightness measures the reflection of 457nm incident light from the substrate surface. Brightness depends on the amount of light scattered or reflected. The higher the scattering or reflection, the brighter the substrate is. This scattering or reflection of incident light increases with an increase in refractive indices of particles either on the surface or in the sheet.

Shown below in Figure 21 and 22 is the influence of size level on the brightness. The sheets made from coarser southern softwood species have lower brightness values that shows the southern softwood species are less bright than the northern species. The size level did not significantly affect brightness. The increase in brightness with the applied coating suggests that the coating significantly contributed to brightness at these coating levels.
Figure 21. Effect of Size Level on Brightness of Southern Species.

Figure 22. Effect of Size Level on Brightness of Northern Species.
Effect of Coarseness, Size Level, and Coating Level on Print Properties

To assess the ink-jet printability of the coated and uncoated papers, the substrates were printed with the piezoelectric Epson Stylus Pro EX ink-jet printer. The print quality was evaluated by measuring print density, delta gloss, and image analysis.

Print Density

The print density was measured with the X-rite densitometer for the 100% black area of printed image of all the samples.

The results of the print density at different size levels and coating levels are shown in Figures 23 and 24. Print density increased with the increasing size level. This is due to the higher ink holdout properties of the heavily sized base sheet that hold the ink in the coating layer rather than allowing it to penetrate into the base sheet. A slight decrease in the density with coating level was observed. This may be due to the penetration ink drop into the depth of coating layer and does not remain on the surface because of sheet's higher absorptivity. The sheets of the coarser softwood species gave higher print density values because of their lower absorbency.
Figure 23. Effect of Size Level on Print Density of Southern Species.

Figure 24. Effect of Size Level on Print Density of Northern Species.

**Delta Gloss**

Paper and print gloss were measured at a 60° angle. The delta gloss is the difference of the print gloss and the paper gloss. Higher (positive) delta gloss means higher print contrast, which is perceived by most consumers to
mean better print quality. Gloss is related to smoothness of the paper surface. Gloss increases with increasing smoothness.

Effect of sizing and coating on the delta gloss is shown in the Figures 25 and 26. Delta gloss is negative for all the substrates. Surface of the sheet tends to fluff when the printing ink is water based. This fluffling of fiber or coating leads to sheet roughening and ultimately reduces the print gloss. And so, delta gloss becomes negative. Coarseness and size level did not significantly affect the delta gloss. Coated samples have higher delta gloss than uncoated samples. Moreover, it increased with the coat weight. The coating layer gives uniform absorptivity that is helpful in producing better print contrast and sharpness.

Figure 25. Effect of Size Level on Delta Gloss of Southern Species.
Figure 26. Effect of Size Level on Delta Gloss of Northern Species.

Image Analysis

Print quality of the coated and uncoated substrates was determined by measuring grid roundness of the print. Since the grids are square, the roundness value for a square should be 1.27 according to the equation below:

\[
\text{Roundness} = \frac{P^2}{4\pi A^2}
\]

Where \( P = \) Perimeter

\( A = \) Area

Therefore, the data were interpreted according to this fact. As the roundness value deviates from this value, it becomes worse.
Roundness

The effect of size and coating level on the grid roundness is shown in Figures 27 and 28. The roundness was higher than 1.27 units for all the samples. The square was better with the coated sheet than the uncoated. And it became slightly better with increasing coat weight. The uniform coating layer gives uniform and ready absorption of successive ink drops that dry fast and result in a sharper image. Coarser species produced poorer roundness values. This may be due to the absorbency effect. Similar results were obtained for the 6 lb/ton sized sheet.

Figure 27. Effect of Size Level on Grid Roundness of Southern Species.
Size Reversion

The secondary objective of this research was to examine the size reversion phenomenon for all the substrates. Size reversion was measured using the Hercules Sizing Tester that is used for measuring absorptivity of the substrate. For measuring size reversion, the HST was measured for all the samples at specific time intervals because size reversion is the natural loss in sizing with time under ambient conditions. Initially, the HST was measured after 9 days from the paper trial runs. The samples were re-measured approximately one month interval and then the HST was measured after three months because the sizing was almost stable at that time.
Size reversion phenomenon is shown in the Figures 29 to 36. All the samples had size reversion with time. Size reversion was not significantly influenced by size level, coating level, and coarseness.

Figure 29. Size Reversion of Uncoated Southern Species.

Figure 30. Size Reversion of Uncoated Northern Species.
Figure 31. Size Reversion of Coated (18% Solids) Southern Species.

Figure 32. Size Reversion of Coated (18% Solids) Northern Species.
Figure 33. Size Reversion of Coated (22% Solids) Southern Species.

Figure 34. Size Reversion of Coated (22% Solids) Northern Species.
Figure 35. Size Reversion of Coated (25% Solids) Southern Species.

Figure 36. Size Reversion of Coated (25% Solids) Northern Species.
CHAPTER VI

CONCLUSIONS

1. The base sheets containing either southern and northern softwood fibers were produced on the pilot plant machine at Western Michigan University. After manufacturing, the sheets were repulped and it was found that compared to northern species, the southern species have 21% higher coarseness. Therefore the coarser southern fiber can be identified in the final sheet by measuring fiber coarseness.

2. The base sheets with four different size levels, 2, 3, 4, and 6 lb/ton AKD, were produced. Absorbency of the base sheet decreased with increasing size level. Size level did not influence paper properties including (a) roughness, (b) porosity, (c) opacity, and (d) brightness. Coat weight decreased with the size level. So, the size level affected the size press pick-up or final coat weight.

3. Base sheets were size press coated with three different coating levels, 18% solid, 22% solid, 25% solid. The sheet coated with 18% solids have the lowest coat weight and the sheets coated with 25% solid have the highest coat weight. Absorbency increased with the coat weight. Coated sheets had
lower roughness, lower porosity, higher opacity, and higher brightness compared to uncoated sheets. Roughness and porosity decreased with the coat weight, while opacity and brightness increased with the coat weight.

4. At the same size level the coarser species had less absorbency. Opacity was not influenced by coarseness. Coarser species had higher PPS porosity, higher PPS roughness, and lower brightness values. Coarser species had less coat weight than northern species.

5. Printing was done using an Epson Stylus Pro EX ink-jet printer. Print density increased with size level. Delta gloss and grid roundness were not significantly influenced by size level.

6. Print density of coated samples was lower and it slightly decreased with coat weight. Delta gloss was higher for coated samples and it increased with coat weight. Grid roundness was also better for coated samples.

7. Print density was higher for sheets made from coarser species. Delta gloss was not significantly influenced by coarseness. Coarser species produced poorer roundness values.

8. All the samples had size reversion with time. Size reversion was not significantly influenced by size level, coating level, and coarseness.
CHAPTER VII

RECOMMENDATIONS FOR FUTURE STUDY

Additional studies using different coating solids is recommended to evaluate the effect of coating solids on the print properties. Also, study using different pigments and different binders (control coatings) will be helpful in determining the effect of coatings used in this research. The same work should be performed with a commercial paper to evaluate the effect of base sheet that was used for this study. The use of species having greater difference in coarseness value is recommended to evaluate the effect of coarseness on paper and print properties. Also, it will be very helpful to print the sheets with more than one ink-jet printers to find out the behavior of different printers for the same substrate.
LITERATURE CITED


Appendix A

Paper Properties Data
Trial Plan for Size Press Ink Jet Coatings

Objective

The objective of this trial is to determine the effects of different levels of internal sheet sizing and coarseness using southern and northern softwood species on the printability of Ink Jet Papers.

Time

Two days on the Pilot Plant Paper Machine.

Materials

1. Hardwood : 60%
2. Softwood : 40% (southern for day 1 & northern for day 2)
3. Sizing Agent : AKD (Hercon 70)
4. Fillers : PCC 200 lb/ton (Albaglos SF dry)
5. Additives : Starch (Stayloc 400) 30 lb/ton

Production

1. Machine Speed : Approx. 100 fpm (operator will set it)
2. Basis Weight : 70 gm/sqm
3. Calendering : 40 psi (3 nips)
Refining

Refine stock using the Double Disc refiner at 60 gpm and 45 kW for 25 minutes. Check freeness. The freeness target is 350 +/- 25 CSF. After checking freeness refine at 1-minute intervals as needed to meet the target.

Run Condition

Follow the attached chart for trial plan. Run in order for simplicity. Run 20 minutes at no sizing, after sheet is ‘STABLE’, run 10 minutes at each size press coating. Run high dryer temperatures.

<table>
<thead>
<tr>
<th>Run #</th>
<th>Sizing (AKD, lb./ton)</th>
<th>Coating Formula</th>
<th>Obtained Coat Weight (gsm)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>Southern</td>
</tr>
<tr>
<td>1</td>
<td>6</td>
<td>Uncoated</td>
<td>-</td>
</tr>
<tr>
<td>2</td>
<td>4</td>
<td>Uncoated</td>
<td>-</td>
</tr>
<tr>
<td>3</td>
<td>3</td>
<td>Uncoated</td>
<td>-</td>
</tr>
<tr>
<td>4</td>
<td>2</td>
<td>Uncoated</td>
<td>-</td>
</tr>
<tr>
<td>5</td>
<td>2</td>
<td>#1 = 18% Solids</td>
<td>6.44</td>
</tr>
<tr>
<td>6</td>
<td>2</td>
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Classification of Dependent and Independent Variables

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Evaluation of Main Effect Variables and Interactions at $\alpha=0.05$

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Appendix B

Print Properties Data
### Classification of Dependent and Independent Variables

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### Evaluation of Main Effect Variables and Interactions at $\alpha=0.05$

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Appendix C

Size Reversion Data
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### Evaluation of Main Effect Variables and Interactions at $\alpha=0.05$

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Appendix D
Print Pattern
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This is printed in Arial 12 pt, which is a sans serif font.
Appendix E

Grid Image From Image Analyzer
Grid Image From Image Analyzer
BIBLIOGRAPHY


