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# Fundamental Study of the Coating and Basesheet Influences on Gloss in Relationship to Coated Inkjet Papers

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# FUNDAMENTAL STUDY OF THE COATING AND BASESHEET INFLUENCES ON GLOSS IN RELATIONSHIP TO COATED INKJET PAPERS

by

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The use of fumed metallic oxides for glossy ink jet media offers alternatives to expensive cast coated and extruded film methods. Fumed metallic oxide pigments provide a high internal porosity and packing porosity that enable the rapid diffusion of liquid inks into the coating layer. Their small particle size (0.1-0.3μm), enable the development of gloss. However, calendering is needed to develop gloss values in the range of cast coated and extruded film grades

Calendering of coated sheet reduces production speed, adds additional handling and rewinding cost (if performed off-line) and reduces the bulk and porosity of the coating layer, consequently reducing the coating's absorptivity. This study focuses on developing gloss without calendering. Factors, which influence gloss such as basesheet roughening, binder level, and smoothness, were examined.

The objectives of the research were to improve the gloss of the coated media through basesheet modifications and coating optimization. The results showed gloss to be significantly influenced by calendering. Significantly higher gloss was obtained for the alumina pigment than the silica pigments. The gloss value of the alumina pigment was found to be comparable to commercially produced papers. The optical properties of the coatings were not influenced by coat weight.

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# CHAPTER I

# INTRODUCTION

Inkjet printing is a printing method where small ink droplets are expelled and made to adhere to the recording medium so as to form dots. During inkjet recording there is little noise, full color images are easily obtained, and, as no developing and fixing is required, recording can be made at high speeds. Inkjet printing has proven to be the first digital printing technology that has achieved an acceptable level of color quality at an affordable price for the majority of home/office end users (1). It is believed that inkjet printing will continue to expand into additional printing markets and will relegate most other digital color printing technologies to niche markets and may begin to challenge electrophotography in many high-end applications. But, at the same time, in line with diversification of needs, there has been a notable increase in the demand for larger image sizes, higher speeds, and high definition of the recorded image for indoor and outdoor applications. Thus, based on these needs, inkjet recording media with the following properties are needed (2):

1. Ink applied to the recording paper must absorb without running and must not Smudge.

2. Ink dots formed on the paper must have high contrast and bright color tone.

3. Ink dots must be suitably distributed in the direction of the paper surface, the dots should be almost round with sharp edges.

4. The paper must have excellent smoothness and gloss so that clean, and bright recorded images are obtained.

Recent research by Lee (3), showed fumed metallic oxide pigments to be capable of producing semi and high gloss ink jet papers with acceptable print quality, after calendering. However, additional studies are needed to optimize his findings and determine the optimal obtainable gloss without calendering.

This investigation focused on the development of glossy inkjet media without calendering. Basesheet modifications, such as, sizing and pre-calendering to improve basesheet smoothness, and coating modifications, such as, various pigment-to-binder ratios, and the use of synthetic pigments were explored.

# CHAPTER II

## LITERATURE REVIEW

Inkjet printing has rapidly become widespread as it produces little noise and makes high speed printing possible (2). Using two or more ink nozzles, color printing can be performed relatively easily by a variety of color inkjet printing systems. Recently, the use of inkjet printers as a means of obtaining hard copies of computer generated images has gained attention as a way to generate quick and accurate color proofs for the printing industry. As digital technology continues to develop, the demand for photographic quality, color inkjet images escalates. Inkjet paper is a special sheet comprised of a porous, ink absorbing layer which can be difficult to dry between color applications when a conventional inkjet media is used for color proofs for which high gloss is required. A porous ink absorbing layer reduces gloss, but a glossy, non-porous layer reduces the aqueous ink's receptivity. As a result, the ink remains wet for a long time on the surface and more time is needed to dry the printed image. Glossy inkjet papers have become increasingly popular, but due to high production costs, are expensive in comparison to other commercial grades of paper.

# Inkjet Paper Types

Inkjet papers are classified into three different categories. The categories are as follows:

## Plain or Uncoated Inkjet Paper

The characteristics of this paper are low cost, good monochrome text, poor color (limited coverage, poor gamut, high bleed, strike-through, cockle/curl). These are inexpensively priced: $0.01/sheet and used for low quality output.

## Matte Coated Inkjet Papers

The characteristics of this paper are relatively high cost, good color gamut, fast drying time, excellent resolution, 100% coverage. These are priced ~$0.10-0.20/sheet and offer the best overall performance (cost basis).

## Glossy Inkjet Papers

The characteristics of this paper are very high cost, non-porous, slow drying time, and poor water fastness. These are priced: $.80/sheet and are capable of producing very high quality color images and are the best papers available for photo quality imaging.

## Trends of Inkjet Recording Papers

The market for non-impact printing has grown rapidly, and inkjet printers are sharing or even leading this development (4). A closer look should be taken at the expression, "inkjet paper," because many paper grades may hide behind this definition. Quality demands are very different depending on the various segments of application. Highest quality in terms of color brilliance, color densities, and dot shapes is required only by end users such as designers or business offices for presentation purposes. These papers, which may also serve as alternative photographic carriers, are at the top of the coated paper pyramid, the base of which are low end office papers for multipurpose use. Coating layers for high-end users are usually applied to wood free papers and consist mostly of silica pigments, and special binders and additives (4).

Glossy inkjet papers are in high demand because they provide photographic print quality, thus, can be used for color proofing in the printing industry.

The various applications of inkjet printing are as follow:

1. Home and office- In the home and office, desktop inkjet printers have made rapid advances in recent years. The convergence of hardware and software technologies has allowed digital imaging and photo realistic printing to become a reality.

2. Industrial – example, bar coding.

3. Wide format – Inkjet printing is replacing traditional printing techniques to create large images in such applications as posters, signage, and billboard printing.

4. Other areas – Including high speed postal addressing, wire cable marking, personal check printing, label printing, and carpet printing.

## Properties of Coating Colors for Inkjet Applications

### Ink Properties

The composition of inks for inkjet printers varies from printing-to-printing system, printing device manufacturers, and time (4). Generally, about 3% to 5% of the ink composition is made from dyes, usually acidic or direct dyes, but sometimes carbon black. The major percentage of the ink is usually water (50 to 90%) and humectants to prevent nozzle clogging and aid in penetration. Additional components of the inks are surfactants to control drop size and shape, complexing agents (water fastness) and electrolytes. Electrolytes are especially needed for continuous inkjet applications, where the droplets are charged before they hit the paper or are captured and recycled during the ejection process.

### Printing Process

Before reviewing the required properties of the inkjet coating formulations and the factors that influence the gloss development of inkjet coated papers, let us briefly review the inkjet printing process.

Depending on the resolution of the printer, the inkjet nozzle ejects a droplet with a size of about 5-40 µm. This droplet contacts the paper and spreads on the surface to a visible dot size, approximately twice the size of the droplet (4). Print quality depends substantially on this spreading and expansion process, with uniform and circular dots providing the best printing results.

Therefore, coated inkjet papers have to exhibit a number of properties to ensure print quality: (a) Controlled and limited expansion of the droplets to dots of perfect edge definition and high ink hold out; (b) Well defined diffusion of the ink on the surface; (c) High absorption rates of the inks; (d) Enhanced color effects in term of gloss, ink density, and color fidelity; and (e) Minimal show-through.

The above properties depend to a high degree on the coating pigments present in the coating layer. For high speed coating applications, high solids are needed to reduce the dryer loads and minimize basesheet roughening during application. The rheology and water retention of high solids coatings is especially important for blade coatings, so as to prevent scratching and streaking. Blade coating provide the smoothest coated surface, and consequently, better gloss.

## Interaction of Inks With Plain Media

For plain papers, hydrogen bonding and van der Waals forces are the key interactions; however, for coated papers and film ionic or Columbic forces are also important (5). Plain and modified papers consist mainly of cellulose and this is what the inks interact with when printed onto the paper surface. For these papers, the furnish, as well as the internal and surface sizing play an important role in determining the print properties. The capillary absorption of the aqueous ink influences how rapidly the ink diffuses into the substrate.

## Interaction of Inks With Coated Media

Coating formulations are a complex mixture of binders, pigments, fixing agents, and optical brighteners. The selection of the components, both the coating and the ink formulation, has a significant influence on the image quality, fastness properties, and rate of ink absorption. The ink vehicle must be rapidly absorbed by the media to yield fast drying times. The diffusion of the colorant into the media must also be carefully controlled to provide the required image quality. The ink receiving layer must also retain its original surface characteristics such as gloss, and avoid distortion or cracking of the coating upon drying. Coating media can contain inorganic oxides such as alumina, silica, talc, clay, titanium dioxide, and calcium carbonate. Polymers such as polyvinyl alcohol, starch, or synthetic latexes are also present to bind the pigments to the paper and each other. The final coating mixture determines the image properties of the print. The interaction of the ink with plain paper and coated paper is contrasted in Figure 1 (5).

To develop the above mentioned properties on an inkjet paper along with gloss, a thorough understanding of gloss and the coating properties which influence gloss is needed. Some of the properties which influence gloss are: coating structure, method of calendering, level and type of binder, and pigment selection.

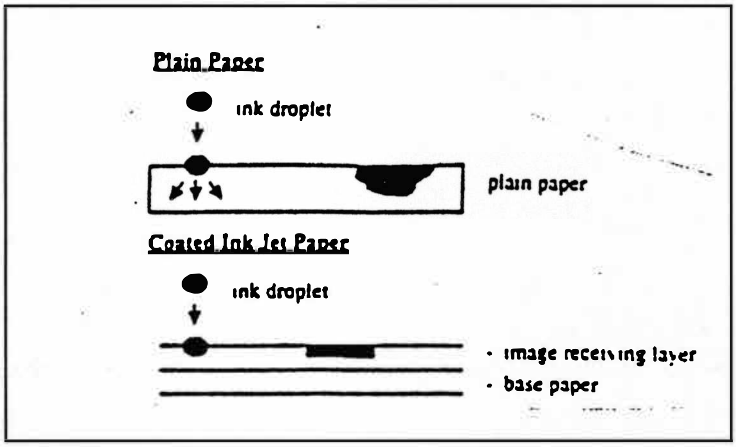


Figure 1. Ink Absorption by Plain and Coated Media (5).

## Gloss

Specular gloss is the degree to which the surface simulates a perfect mirror in its capacity to specularly reflect incident light. Specular reflection refers to the portion of incident light which is reflected from the surface of an object with an angle of reflection being equal to the angle of incidence. The amount of specular reflection is determined by: (a) The index of refraction and size of the pigment particles, (b) The wavelength of the incident light, and (c) The angle of incidence.

Parallel beams of light striking an optically flat surface at the same angle of incidence will be reflected as parallel beams. The type of reflection that occurs on a non-flat surface is called diffuse scattering. Surfaces can be made flat by either calendering or filling of the valleys with pigments. The different types of reflections and light scattering mechanisms are shown in Figures 2 and 3, respectively.

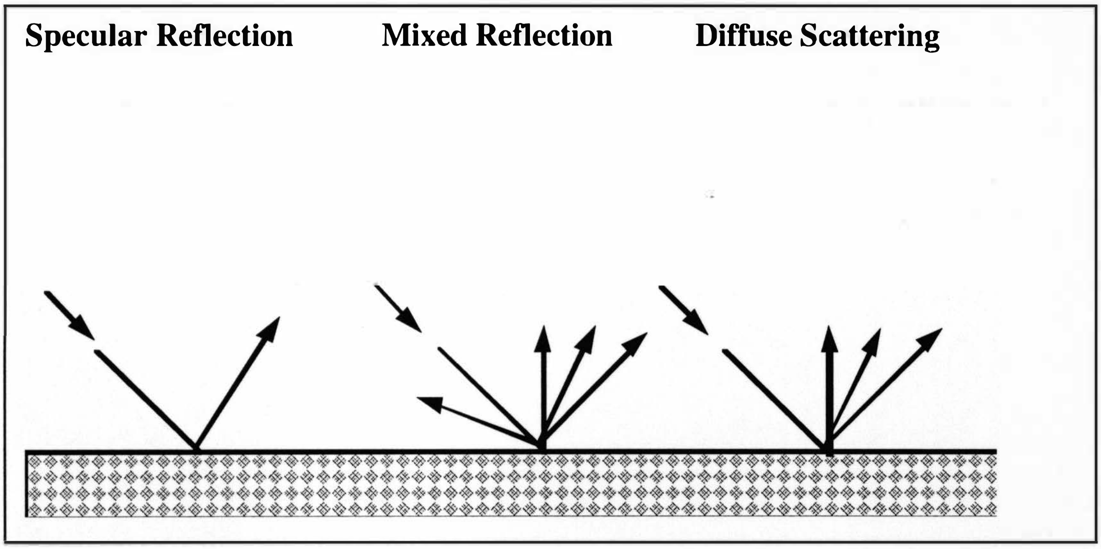


Figure 2. Types of Light Reflection.

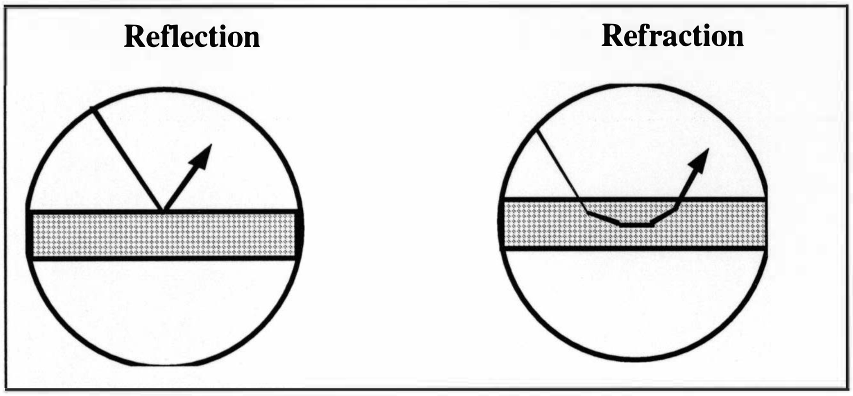


Figure 3. Mechanisms of Light Scatter.

Paper Gloss Measurements

The two specular angles which are most commonly used in the paper industry to measure gloss are 75 degrees and 20 degrees (measured with respect to a perpendicular to the surface of the sheet). These measurement angles were selected based on perceptual studies which showed these angles to best correlate measured gloss values: (a) 75 degree gloss – for low to medium gloss papers, (b) 20 degree gloss – for high gloss papers (greater than 80% reflectance at 75 °).

## Coating Structure Influence on Gloss

According to Lee (6), the gloss of paper coatings are mainly affected by the following factors:

1. The effect of particle size and shape. Coating gloss decreases with increasing particle size.

2. The effect of particle size distribution. Since small particles increase the packing efficiency of particles by filling the void spaces, it is obvious that coating gloss can be improved by increasing the fraction of small size particles.

3. The effect of colloidal stability. The flocculation or destabilization of suspended particles leads to a porous sediment or coating. It has been noted that the gloss of certain coatings reaches a maximum at the point of optimum destabilization and then decreases with further addition of destabilizers. Ammonium sulfate and calcium chloride are examples of some destabilizing agents.

4. The effect of type and binder level. Previous work by Lee (6), showed the gloss of coatings to be influenced by the amount and type of binder used. From his results, Lee concluded that shrinkage of the binder during drying, roughens the surface of the coating layer, causing the gloss to decrease. The amount of surface

roughening experienced during drying depends on the type and amount of binder used in the coating formulation.

5. The effect of drying conditions. Although there is no significant influence of drying conditions on the gloss of low temperature film forming polymer coatings, the gloss of high temperature film forming polymer coatings are strongly dependent upon the drying conditions. It can be seen that the gloss of high temperature film forming polymer coatings decreases with an increase in drying temperature.

6. Effect of binder composition. Binder composition also has an influence on the gloss of the coatings in certain cases. For example the ratio of styrene butadiene level in SBR latex binders has an influence on gloss. It has been noted that increasing the level of styrene in SBR latex increases the hardness of the binder which prevents the complete coalescence of the latex particles during drying. This results in less shrinkage, hence higher gloss, but occurs at the expense of binding strength

## Pigment Particle Porosity and Its Influence on Gloss

In an inkjet recording sheet it is common to provide an ink receiving layer employing a porous pigment with an ink absorbing property suitable for controlling the color effect and dot definition which are decisive for image quality (7). The many voids of the ink-receiving layer scatters light, preventing the transmittance of light, causing the ink-receiving layer to be opaque. As a result, the image tends to be whitened, and the color reproducibility and density tend to be low because it is difficult for the light to reach the ink colorants located in the internal voids of the pigment and coating layer. Since the high porosity of the ink-receiving layer tends to reduce the specular reflection of light, it is difficult to achieve gloss with these pigments.

## Inkjet Coating Pigments

High structure amorphous silica pigments are used in the paper industry, especially for ink jet papers. In this application, they provide better ink receptivity, brightness, opacity, bulk, and oil and water absorption values than conventional coating pigments, i.e., clay and carbonates. Amorphous silicas are classified as colloidal, fumed, precipitated and gelled

Colloidal silica is a stable dispersion of non-porous particles in water. These particles are termed primary particles because they are composed of dense-phase silica which can range in size from roughly 10 nm to over 100 nm. Porosity in systems comprised of colloidal silica arises from the packing of the particles. Surface area is a direct measure of the external surface of the spherical particles and hence is related to particle diameter.

Fumed, pyrogenic silica is produced by the flame hydrolysis of silica tetrachloride or tetrafluoride. The result is an aerosol of small primary particles typically 7-40 nm in diameter. These particles stick together by flame reaction and sintering to form aggregates, and the aggregates form into micron-sized agglomerates. Porosity is created by virtue of the packing of the dense particles into secondary structures. These secondary particles are shear insensitive, thus shear will not destroy the inherent porosity which gives them an inherent absorptivity. Precipitated silicas are conceptually similar to fumed silicas, only the primary particles are precipitated from solution. Fumed and precipitated silica agglomerates, thus resemble a cluster of grapes on a molecular scale. Silica gels are unique in that they consist of small primary particles (25 Angstroms) which are welded together chemically to give a three dimensional structure that is inherently porous. They are produced through an agglomeration process under conditions where the silanol groups on the surface of the small primary particles come together and condense to form siloxane bonds. As a result of this polymer isolation process, a three dimensional network is formed. The network entrains water, yielding rigid gel like materials having a large internal porosity. The network is then aged and dried to maximize the formation of the siloxane bonds. After aging, the structure resembles a sponge on a molecular scale.

For inkjet applications, the internal porosity of silica particles, as well as packing density, effect print quality and dry time. Pores between the silica particles allow for the rapid diffusion of the ink into the coating structure while also providing a capacity for liquid uptake. In addition, the silica displays a partitioning effect in which ink liquid is preferentially absorbed into the porous structure leaving the dye colorant molecules near the coating surface. These two effects allow high optical densities to be achieved. Studies performed by Lee (3) showed the influence of particle size, pH, pigment type, coat weight, and binder on the print quality of glossy inkjet media. Lee's work showed a significant improvement in gloss, with calendering for fumed metallic pigments, but little improvement for gelled silica.

From his work, Lee concluded that the particle size and shape of the fumed metallic oxides studied were capable of producing a high glossing inkjet coating, with acceptable print quality, after calendering. Lee also noted that the silica/polyvinyl alcohol ink jet coatings are limited to low solids, which promotes basesheet roughening due to the penetration of coating water into the base sheet during application and metering.

## Glazing of Paper

The term glazing refers to producing gloss on the web surface. The most common surface glazing processes can be described as follows: (a) Machine calendering, (b) Supercalendering, (c) Gloss calendering, and (d) Soft Nip and Hot/Soft Nip calendering.

In machine calendering surface directional forces are not applied and therefore the term glazing is not used. The aim of glazing of coated papers is mainly to improve paper printability (printing smoothness) and appearance (gloss). These desirable properties are, however, reached at the expense of paper strength, stiffness and opacity.

In general, the ratio of the fiber thickness to the pigment, and binder particle size is 100/10/1. Glazing alone cannot produce all the end product qualities desired by the printer. For example, glazing will improve the gloss and smoothness of a sheet, but will adversely affect the brightness and ink receptivity of the sheet. Glazing also reduces the bulk and stiffness of the sheet. Thus, the properties of the basesheet and the coating must be negotiated to obtain the desired surface properties.

The internal structure of the coating layer has traditionally been manipulated by altering the pigment size distribution, shape and orientation of the pigments. Since, however, the ingredients generally used in the coating color do not have essential differences in the index of refraction, the reflection of light mainly occurs on the air boundary surfaces. Therefore, changes in most conventional coating ingredients will not contribute to larger differences in light scattering. Decisive factors are the size, form and quantity of air pores. Indeed, this mode of thinking is more descriptive from the point of view of glazing, since glazing can hardly have an effect on the size of the pigment itself, but it does have an effect on the pores. Similarly, such a mode of thinking emphasizes the fact that in addition to glazing, the coating structure before glazing is essential from the point of view of the final result. In addition to the coating color ingredients and the base paper, the coating process itself and the coating color drying conditions have an effect on the final coating structure. The internal structure of a well glazeable coating is as porous as possible. A narrow pigment size distribution results in best sheet coverage. If the number of pores is high and the pores are small in size, more light refracting surfaces are obtained. To produce light scattering, these pores have, however, a certain optimum size, which is close to ½ the wavelength of visible light. The result of the porosity requirement is also that a good coating color is not packed into the base paper pores.

The coating surface has to be as level as possible to obtain a good smoothness. As for gloss, the levelness requirement alone is not sufficient, but furthermore, the asymmetric particles on the surface have to be approximately parallel to the surface. To obtain suitable sorption properties, the coated surface has to be either compact or microporous, depending on the printing method. It is difficult to produce a surface with the above properties by coating the surface only once, i.e. a surface which is internally porous and disordered, as well as, compact and oriented on the surface. Because of this glazing is an essential part of coated paper manufacture. In some rare cases, however, a cast coating method is used, in which the coating color is dried against a polished chrome cylinder. In this process, the cylinder surface is replicated onto the coated paper so calendering is not required.

## Development of Smoothness and Gloss

This aspect is viewed mainly from the point of view of the web, and not so much from the point of view of the process and process equipment. There are four factors affecting the development of smoothness and gloss: (1) Compression of the whole web, (2) Substance transference and removal, (3) Particle orientation, and (4) Replication of the glazing surface.

Although the compression and substance transference are essential for the improvement to surface smoothness, the build-up of gloss is more affected by the orientation of asymmetric particles at the surface. The asymmetric orientation of pigment particles results from the replication of the calender roll surface onto the surface of the coated substrate.

Calendering affects the exposed surface in a manner that depends largely on what is happening inside the sheet (8). Where there is no coating, the outermost fibers replicate the roll surface reasonably well. At high temperatures, lateral flow of the fibers can be seen. Irregularities of the sheet surface affect the properties of the sheet in two different ways. First, on a microscopic scale and low roughness level, the irregularities are responsible for the specular gloss. Second, at the size of the fiber flocks and shives, the irregularities determine the smoothness as measured by air leak methods. The calender will change these irregularities to different extents.

Considering the different types of calenders available in the market today. It can be seen (9) that hot/soft nip calendering offer various advantages over other hard calendering methods. Some of the advantages are as follows: (a) Better printability due to more coating porosity and better ink receptivity, (b) More uniform densification, hence more uniform ink receptivity, (c) More uniform compressibility, hence more uniform ink transfer, (d) Higher printed gloss, (e) Better paper uniformity like less gloss variation, reduced blackening tendency, reduced barring tendency, and (f) On-line operation.

Considering the fact that soft nip calendering is well developed and is increasing in use, it looks very attractive to be used in inkjet base sheet calendering applications.

# CHAPTER III

## STATEMENT OF PROBLEMS AND OBJECTIVES

Although coatings formulated with fumed metallic oxide pigments have proved to be capable of producing glossy inkjet media, calendering was necessary to achieve the desired gloss values. The need for calendering has the following undesirable outcomes: (a) Additional capital expenditure and maintenance cost, and (b) Reduction of the absorptivity of the coated layer.

In this study, fumed metallic oxide pigments will be used in inkjet coating formulations. Of special interest is whether the use of fumed metallic oxide pigment coatings can provide gloss values comparable to commercially produced glossy ink jet papers with limited or no calendering. The effect of base sheet absorptivity on gloss will be studied.

The goal of this research was to determine if the coating gloss of the metallic oxide pigments, first explored by Lee (3), could be improved by altering the coating and basesheet properties to reduce basesheet roughening upon drying.

CHAPTER IV

# EXPERIMENTAL DESIGN AND METHODOLOGY

The experimental design used during this study was to investigate the influence of various pigment types on the optical and surface properties of the coated substrate at various levels of solids and coat weights. The experimentation was split into four phases: (1) drawdown coating studies, (2) characterization of the optical and surface properties of coated papers and the selection of pigment to binder ratios based on the critical pigment volume concentration curve, (3) application of selected coatings using a cylindrical laboratory blade coater (CLC - 6000), and, (4) characterization of the optical properties, print properties, and dry coating structure, before and after hot/soft nip calendering, of CLC produced samples. The schematic representation of the experimentation is shown in Figure 4.

The properties of the base paper were measured. The base paper was determined to be a 77.5 g/m$^2$ uncoated inkjet paper from Wassau Papers (Otis, Maine). Selected pigments were obtained from the sponsoring company. Three different silicas (A, B and C) of varying surface areas, size and shapes and one aluminum oxide (D) pigment with a surface area similar to the pigment A was obtained. The physical properties of pigments are shown in Table 1.

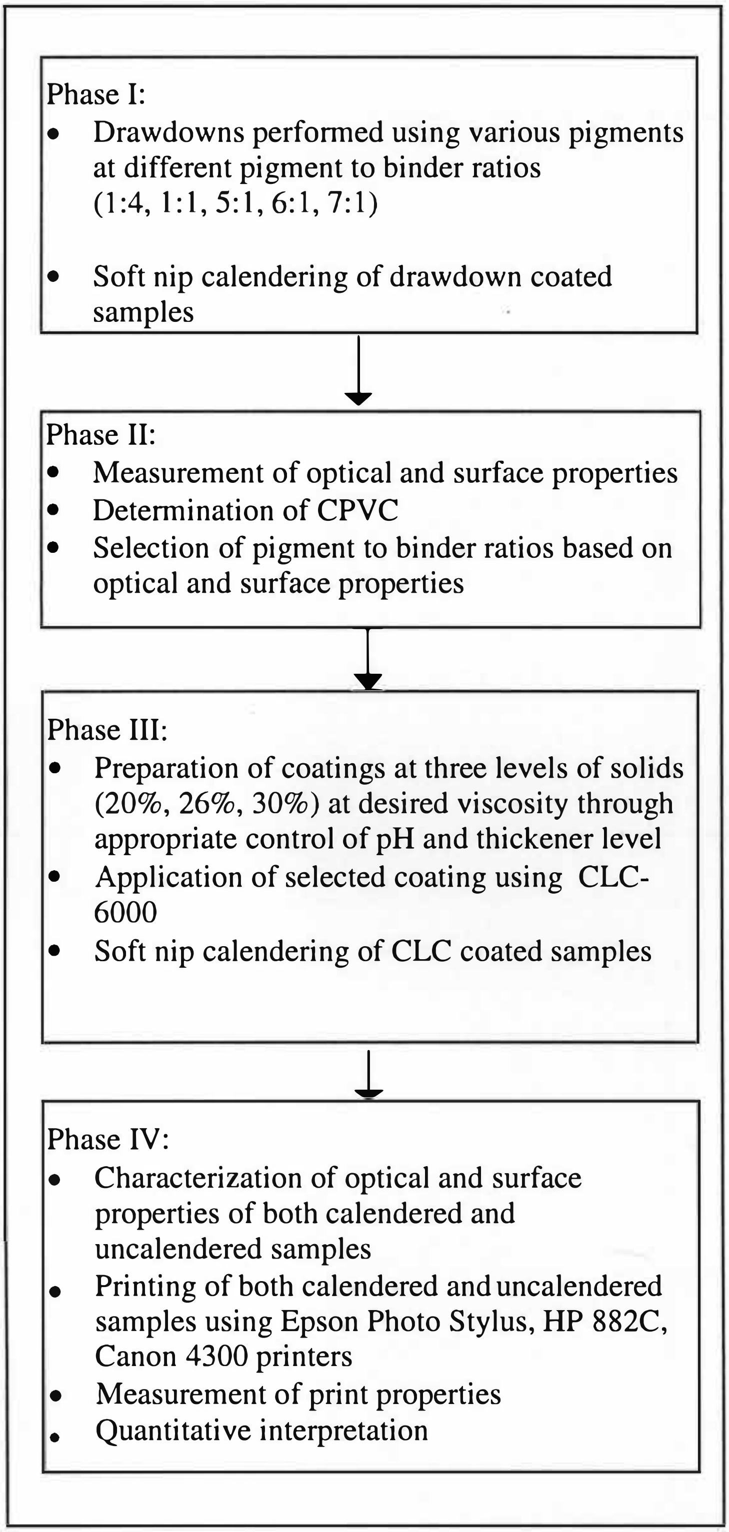


Figure 4. Schematic Representation of Various Experimentation Phases.

Table 1

The Physical Properties of Pigments as Supplied

| Sample | Solids Content % | Color | Specific Gravity ($g/cm^3$, 25° C) | pH |
|---|---|---|---|---|
| Fumed Silica 019(A) | 25 | White | 1.2 | 9-10 |
| Fumed Silica 020(B) | 30 | White | 1.2 | 9-10 |
| Fumed Silica 001(C) | 30 | White | 1.2 | 10-10.3 |
| Aluminum Oxide 003(D) | 40 | White | 1.4 | 3.8-4.2 |

The binder used in the coating formulation was a partially hydrolyzed and low viscosity polyvinyl alcohol (Airvol 203, Air Products Inc.). This polyvinyl alcohol was chosen to optimize the % coating solids by minimizing the interaction between pigments and PVOH. Polyvinyl alcohol with a higher degree of hydrolysis are known to interact more strongly with the pigments, thus limiting the % coating solids which can be feasibly prepared. Solutions of polyvinyl alcohol were prepared at 30% solids by adding the required amount of dry PVOH powder to cold water and defoamer (Foam Master VF, Henkel, Inc) under agitation and heating the mixture to 185°F. The solution was held at this temperature for 35-40 minutes to assure complete dissolution and hydration of the PVOH. The solids content of the solution was measured using a CEM Labwave 9000 microwave moisture analyzer. The solution was cooled to 140°F before adding the slurried pigments at a slow rate of agitation. The coatings were mixed for 20-30 minutes and the pH and viscosity were measured.

Coatings were prepared using the supplied pigments and PVOH binder at five different pigment to binder ratios (1:4, 1:1, 5:1, 6:1, 7:1). The viscosities of the coatings were measured using a Brookfield RVT digital viscometer (#4 @ 100 rpm). If the viscosity of the coating was too high to mix or apply, adjustments were made by adding a little distilled water to the coating mix and the final coating solids were then re-measured.

## Drawdowns

The coatings were applied to the uncoated base paper using several different Meyer rods. The Meyer rods were selected to enable 4-12 $g/m^2$ coating to be applied. The samples were allowed to air dry overnight. The samplers were then cut into 5-inch diameter circles using a press punch. The coat weights were then determined, taking the difference between weights of the uncoated and coated samples after being dried in the CEM Labwave 9000 microwave moisture analyzer. The samples were then conditioned according to TAPPI standard test methods before performing optical and physical property measurements. The brightness of the papers was measured using a Technidyne brightness meter, TAPPI procedure T 452 om-92. Gloss was measured using a Hunter75$^{\circ}$ gloss meter according to TAPPI procedure T 480 om-92. The surface smoothness and porosity of the sheets were measured using a Parker Print Surf (PPS) tester (TAPPI T 555 pm-94). The rate of liquid absorption of the papers was measured using a First Ten Angstrom Dynamic Contact Angle measuring device. After reviewing the results of the drawdowns studies by analyzing the Critical Pigment Volume Concentration (CPVC), the pigment to binder ratios for various pigments were selected for additional CLC studies. CLC studies were performed to obtain larger dimensional samples for printing studies and to optimize the surface smoothness of the samples by using a blade applicator and infrared drying under tension.

## Cylindrical Laboratory Coater (CLC) Studies

The base papers were blade coated using a Cylindrical Laboratory Coater (CLC) at a speed of 3000 fpm. Coatings were prepared using three fumed silicas of varying surface properties and an aluminum oxide pigment.

Both pigment slurries and binder solution were adjusted for their pH to the same level to avoid undesirable interactions which caused the coatings to gell under agitation and shear. The coatings were prepared by adding the binder to the pigments under agitation followed by the thickener. The pH of the coating was continuously monitored in order to maintain the pH to level at which the coatings remained stable (did not gel). It was found that the pH of the PVOH binder had to be adjusted to the pH of the pigment slurry to avoid gelling. Because of the low viscosity of these coatings, a measured level of thickener (HEC/CMC) was added to reach the desired viscosity of 600-900cps. During the CLC trials, the samples were pre-dried at 25% power for 10 seconds and post dried at 100% power for 60 seconds. Four different coat weights ranging from 5 - 15g/m$^2$ were applied.

The brightness, gloss, smoothness, porosity and dynamic contact angle of the coated papers were once again measured. The samples were printed on three different inkjet printers; Epson Stylus Pro, Hewlett Packard 882C, and Canon Bubble Jet 4300, using a proprietary test print pattern created with ADOBE software. The print density (solid black areas), print gloss (solid black area) and dot gain (10% black area) of the samples were then measured. Print density and dot gain were measured using an X-Rite 408 densitometer and print gloss was measured using a Gardner 60° micro-gloss meter.

The CLC samples were calendered through three nips using alternating the coated side against the drum. The coated side contacted the metal cylinder first. The calender was set at 6 pli and 60° C. The optical, surface and printing properties were measured and compared to samples before calendering.

# CHAPTER V

# RESULTS AND DISCUSSION

The main objective of the phase I experimentation was to study the effect of pigment to binder ratios on the optical and surface characteristics of the various coated substrates and to determine their critical pigment volume concentration for CLC runs. The properties of the base paper are shown in Table 2.

Table 2

Characterization of Base Paper Properties

| Properties | Value |
|---|---|
| pH | 6.6 |
| Ash | 8.31% |
| Caliper | 0.00362 inch |
| Brightness | 82.7% |
| Gloss | 6.4% |
| Smoothness | 3.93 microns |
| Hercules Sizing test | 109 sec |
| Porosity | 2.77 ml/min |

Phase I and Phase II

The objective of the phase one study was to determine the influence of coat weight, pigment to binder ratios, particle functionality and particle and surface properties of the coating layer.

The properties of the coatings for drawdowns were measured prior to the application. The viscosities, solids and pH of the coatings are shown in Table 3. The viscosities of the coatings were measured with #4 spindle @ 100 rpm on the Brookfield viscometer.

Table 3

Properties of Coatings Before Drawdown Application

| Pigment Type | Pigment:Binder Ratio | Brookfield Viscosity cps | Solids (%) | pH |
|---|---|---|---|---|
| 003 | 1:4 | 1540 | 26.0 | 5.8 |
| 003 | 1:1 | 646 | 29.5 | 5.4 |
| 003 | 5:1 | 129 | 36.7 | 4.4 |
| 003 | 6:1 | 103 | 37.2 | 4.4 |
| 003 | 7:1 | 98 | 37.3 | 4.3 |
| 001 | 1:4 | 1493 | 25.7 | 8.3 |
| 001 | 1:1 | 1200 | 29.4 | 8.3 |
| 001 | 5:1 | 400 | 29.9 | 8.5 |
| 001 | 6:1 | 305 | 30.2 | 8.6 |
| 001 | 7:1 | 218 | 29.8 | 8.6 |
| 019 | 1:4 | 988 | 25.5 | 9.1 |

Table 3 --continued

| Pigment Type | Pigment:Binder Ratio | Brookfield Viscosity cps | Solids (%) | pH |
|---|---|---|---|---|
| 019 | 1:1 | 692 | 27.33 | 9.3 |
| 019 | 5:1 | 240 | 25.8 | 9.2 |
| 019 | 6:1 | 147 | 25.6 | 9.1 |
| 019 | 7:1 | 130 | 25.5 | 9.1 |

By using various Meyer rods, coat weights ranging from 4-18 g/m$^2$ were obtained. The influence of pigment to binder ratios at 10 g/m$^2$ coat weight for various pigments of both calendered and uncalendered drawdown samples are shown in Figure 5 and Figure 6 respectively.

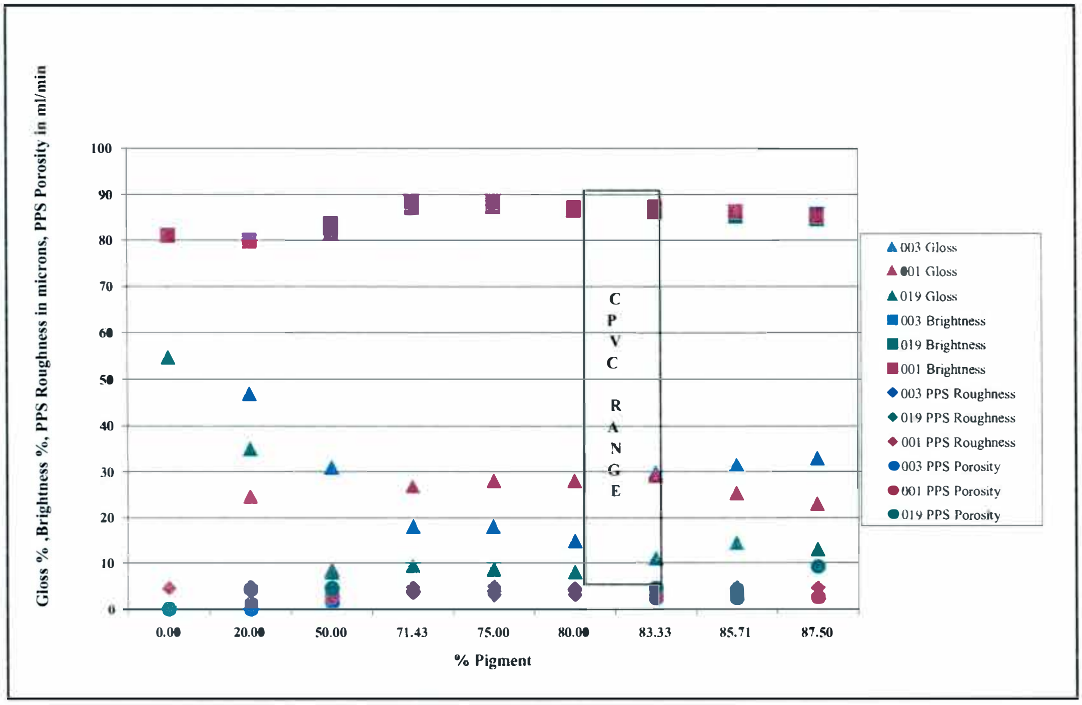


Figure 5. Influence of Pigment to Binder Ratio on Optical and Surface Properties for All Pigments at 10g/m$^2$ Coat Weight on Uncalendered Drawdowns.

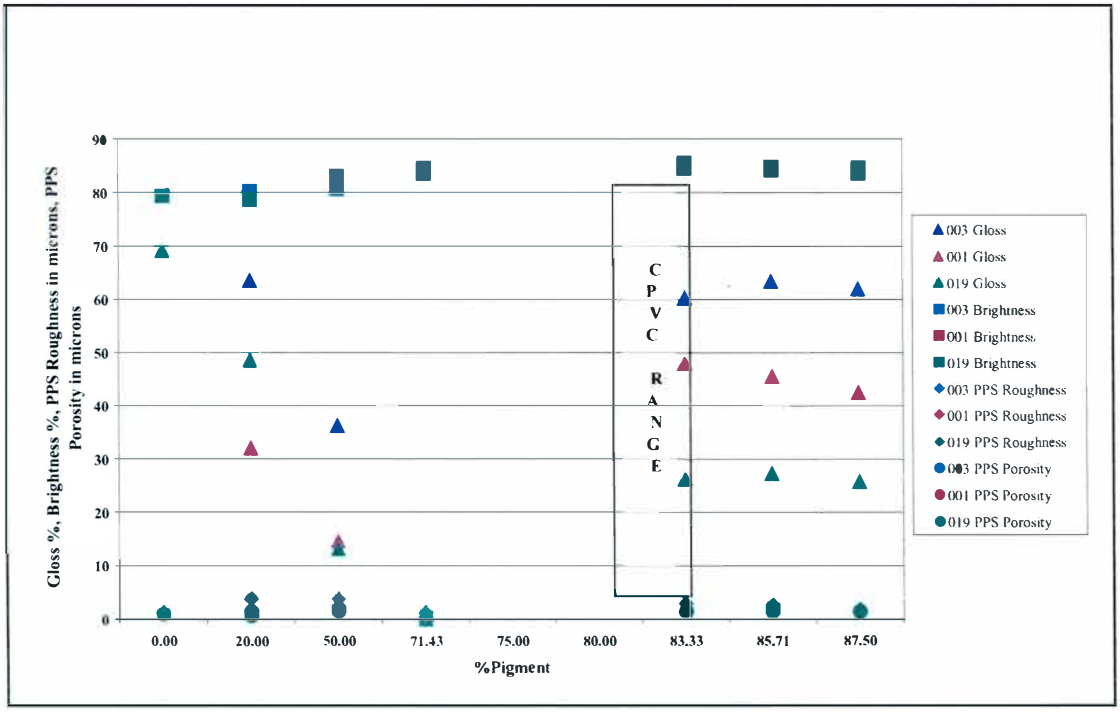


Figure 6. Influence of Pigment to Binder Ratio on Optical and Surface Properties for All Pigments at 10 $g/m^2$ Coat Weight for Calendered (2 nips) Drawdowns .

Coat weight and pigment to binder ratio had an influence on the brightness and gloss of all the coatings. Brightness values were lowest at the higher levels of binder addition, while gloss significantly improved at the higher levels of binder addition. The high gloss values at the high binder levels, results from the presence of a smooth transparent latex film at these points. On the other hand, brightness suffers because insufficient pigment particles are present to scatter and absorb the light.

As the level of binder decreased, gloss decreased due to the disruption of the latex film by the pigment particles, and insufficient scattering of the light by the pigment particles. The U-shaped curves in Figures 5 and 6 indicate there is a significant decrease in gloss, and increase in roughness, around the CPVC (Critical Pigment Volume Concentration) point. The CPVC point (Figure 7) is the point of pigment concentration at which sufficient pigment to air interfaces are present to cause the optical properties of the coatings to improve. Gloss increases above the CPVC because the pigment concentration is sufficient to scatter sufficient light in specular reflection. As the concentration of pigment continues to increase above the CPVC, the pigment particles continue to pack more tightly, resulting in more scatter, hence higher gloss. Figures 5 and 6 indicate that a pigment-to-binder ratio of 5:1 was best for all the coatings because the gloss did not significantly improve above this point. Comparison of the different silica types showed the fumed silica 019 coating to produce less gloss than silica 001. This is attributed to the difference in primary particle size of the fumed silica 019. Fumed alumina exhibits a gloss value as high as fumed silica 001. This may be attributed to the pigment particle type and also its primary particle size is almost equal to that of fumed silica 001.

Calendering had a significant influence on the gloss of all the coatings due to the alignment of the pigment particles (increased in surface smoothness).

## Phase III and Phase IV

After reviewing the results of the preliminary draw down studies, it was decided to prepare coatings for the CLC trials using a pigment to binder ratio of 5:1. This ratio was chosen because the optical and surface properties of the coatings favored these conditions. For the CLC runs, three different solids level were chosen ranging from 20% to 30% for all the silica coatings and 26 % to 33% for the aluminum oxide coatings. This was done in order to observe the effect of base sheet roughening, due to coating water penetration, on the optical and surface properties of

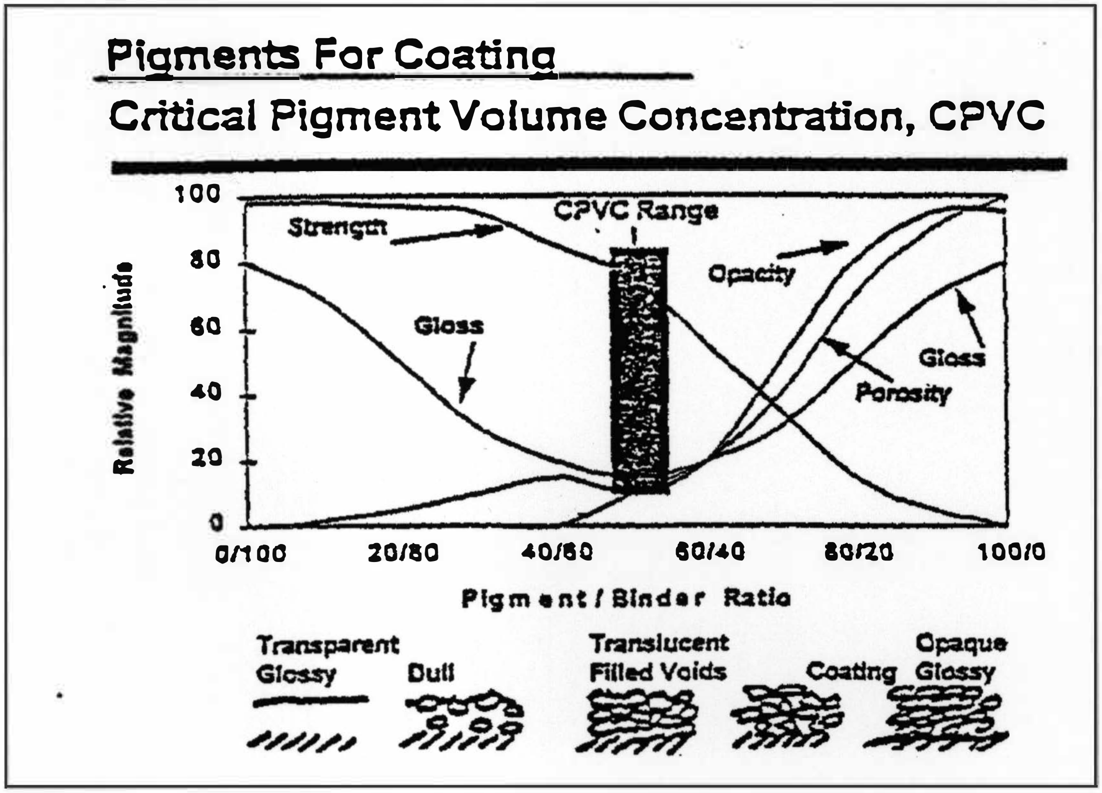


Figure 7.Critical Pigment Volume Concentration for Pigmented Coatings (9).

the coatings. Higher coating solids were obtained for the aluminum oxide coatings because the pigment slurries were supplied at higher solids. The coatings were blade coated at 3000 fpm on the CLC 6000 coater. Coat weights ranging 4-16 g/m$^2$ were obtained. In preparing the coatings, it was found that the at the higher solids levels, the coatings were unstable and gelled if the pH of the coatings was not properly controlled. It was also determined that the CMC thickener could not be used for the alumina pigment due to gelling. For this reason the thickener type was switched to HEC which is nonionic in nature, hence less reactive.

## Experimental Data Analysis

Data analysis was performed using SAS, a statistical software package, to analyze the collected data. In performing the analysis, the GLM (General Linear Model) procedure was used instead of ANOVA. GLM produces ANOVA tables, but handles missing data points more rigorously than ANOVA. Duncan's multiple range test was used to observe the differences between means for effects determined to be significant. The 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 are compared to determine if any statistical significance exists between one treatment and another in a given effect. Data points were collected for various properties by doing at least five replicate measurements. No duplicate measurements were performed.

A statistical analysis was first performed to classify the dependent and independent variables. A GLM model was performed using the independent variables to identify the significant dependent variables by their Pr-values. Choosing the variable having the highest F-Value enable the most significant dependent variables to be identified. The analysis was performed one response variable at a time, eliminating the "printing" response variables and the variables related to the printer type used, initially.

The dependent variables and their interaction with the response variables were also performed. The dominating dependent variable was then removed and the analysis repeated with the other dependent variables to evaluate their individual contributions to the effects. A Duncan's comparison of means test was then used to evaluate the significance of the dependent variables for a given set of experimental conditions. This test shows if any real significance exists between the means of the main effect (dependent) variables evaluated.

The significant dependent variables were then eliminated, and the GLM model re-run to evaluate the interactions of the other, less significant, dependent variables to the printing response variables. A Duncan comparison of means test was again performed.

The GLM model, without printing variables, demonstrated that calendering, pigment type, and solids significantly influenced the optical and surface properties of the coating, respectively. Coat weight did not play a significant role in influencing these properties (Table 6 of Appendix A).

For the uncalendered samples, pigment type and solids significantly altered the brightness, roughness and gloss of the samples (Table 7 of Appendix A). Comparison of the calendered and uncalendered samples shows the uncalendered the brightness of the 001 and 003 pigments to be highest at the 26% solids level showed. For the calendered samples, both solids and coat weight significantly influenced the brightness of the 019 and 020 coatings. The brightness of the 019 coating was the highest for the 11 $g/m^2$ paper coated at 26% solid . For the 020 coating, the highest brightness values were obtained when the coating was applied at the 20% solids level. The decrease in brightness with coating solids for all the pigments is attributed to poor pigment dispersion due to the interactivity of the pigments with the hydroxyl groups on the PVOH, which caused the coatings to gel. The Duncan's grouping also indicated that the properties of the fumed silicas were significantly different than the aluminum oxide pigment

In regards to gloss, smoothness and coating porosity, the most significant main effect variables were calendering and pigment type. A Duncan's comparison of means showed calendering to be the most significant main effect for all properties tested. The aluminum oxide pigment produced the glossiest coating. The 019 fumed silica resulted in the lowest gloss coating. The results from the statistical analysis of these variables are shown in Table 6 of Appendix A.

Once the influence of calendering and coating variables was statistically analyzed, additional statistical analyzes were performed to determine the influence of printer type on print quality. Print quality was characterized by measuring the print density, print gloss, and dot gain of the printed images. From these analyses, it was determined that the coating solids and pigment type most significantly influenced print density and dot gain, while coat weight influenced print gloss (See Tables 6-8 of Appendix A).

Removing the impact of printer type, the effects of pigment type and solids on printing properties were determined. For the HP printer, pigment type and solids significantly altered the print density and dot gain while coat weight had an influence on print gloss. For the Canon printer, pigment type and solids significantly altered the print density and dot gain, but coat weight did not significantly influence these properties. For the Epson printer, only pigment type significantly altered the print density and dot gain.

## Influence of Roughness on Gloss for Various Pigments

Figures 8 and 9 show the relationship between coating roughness and gloss for the alumina and silica coatings before and after calendering. The graphs show the 019 to be rougher. As a result, it produced less gloss. For all the samples tested, calendering reduced the roughness and, consequently, improved the gloss of the coatings.

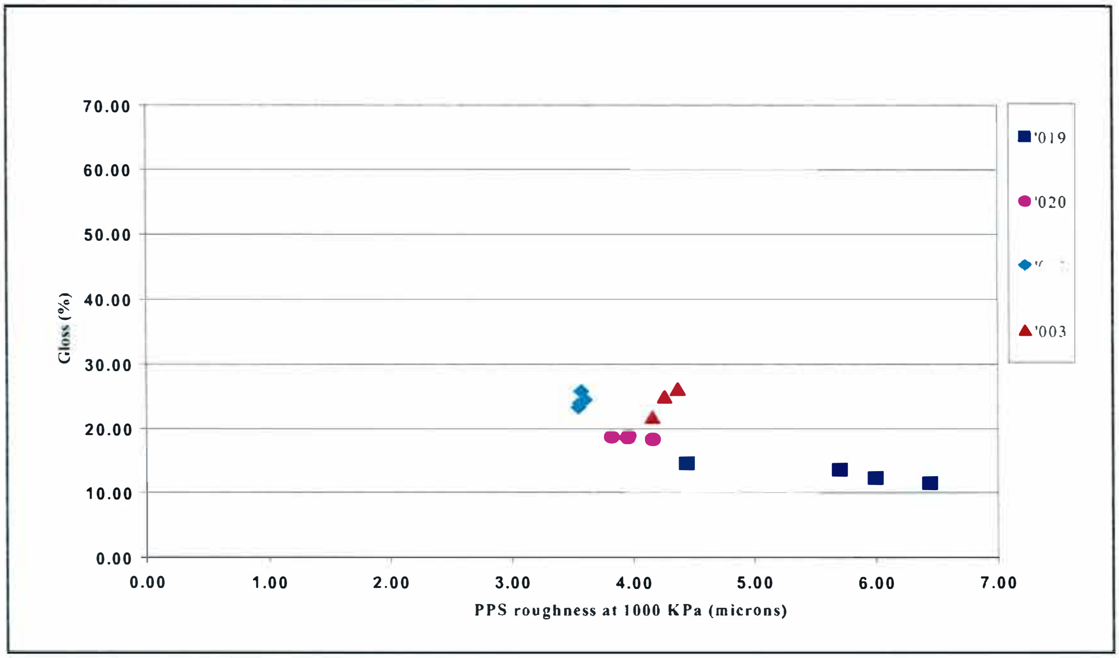


Figure 8. Influence of Roughness on 75$^{o}$ Gloss of Coatings, Before Calendering, All Coatings Were Applied at 26% Solids.

Figure 10 shows the influence of coat weight on roughness, before and after calendering. It is obvious from the graph that calendering significantly improved the gloss. It is also observed for the 019 pigment that the coating roughness increased with coat weight. This is attributed to the poor stability of this coating. It was observed that this coating began to gel as it was being applied. It is believed that as the coating was being applied, the pH of the coating changed due to the evaporation of the pH modifier. As a result, it gelled under the blade causing the pigments to aggregate and the roughness of the coating layer to increase. Additional studies are needed to better understand these phenomena.

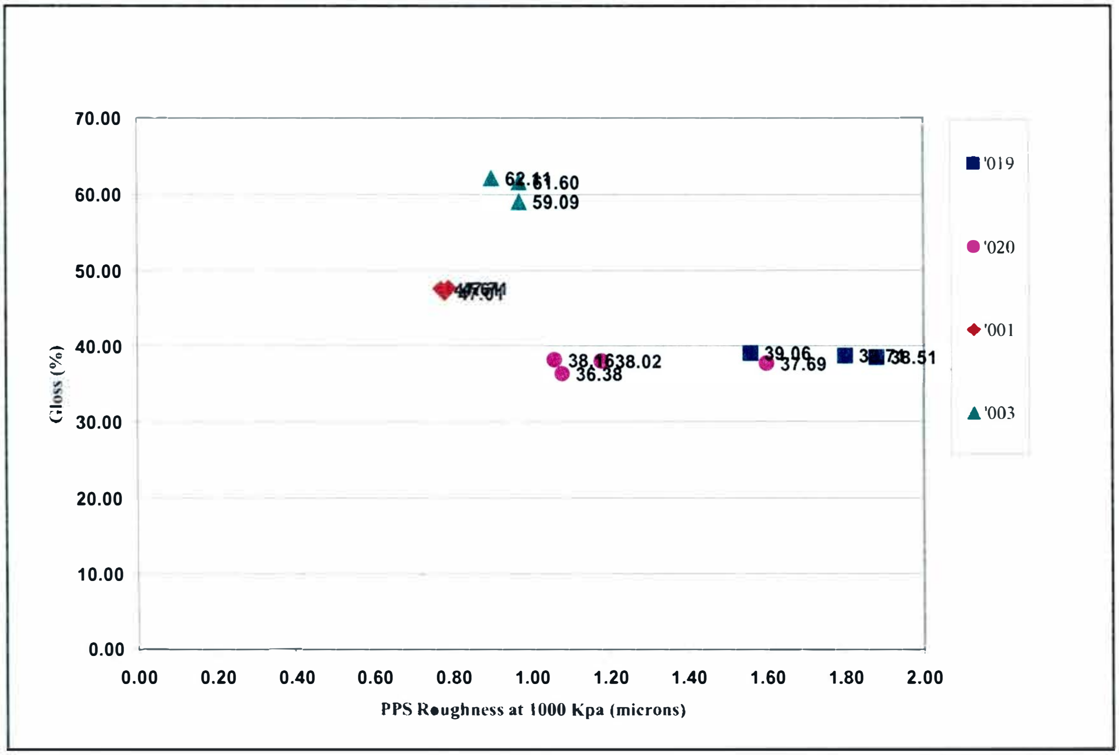


Figure 9. Influence of Roughness on 75° Gloss of Coatings, After Calendering, All Coatings Were Applied at 26% Solids.

Figure 10 shows the influence of coat weight on roughness, before and after calendering. It is obvious from the graph that calendering significantly improved the gloss. It is also observed for the 019 pigment that the coating roughness increased with coat weight. This is attributed to the poor stability of this coating. It was observed that this coating began to gel as it was being applied. It is believed that as the coating was being applied, the pH of the coating changed due to the evaporation of the pH modifier. As a result, it gelled under the blade causing the pigments to aggregate and the roughness of the coating layer to increase. Additional studies are needed to better understand these phenomena.

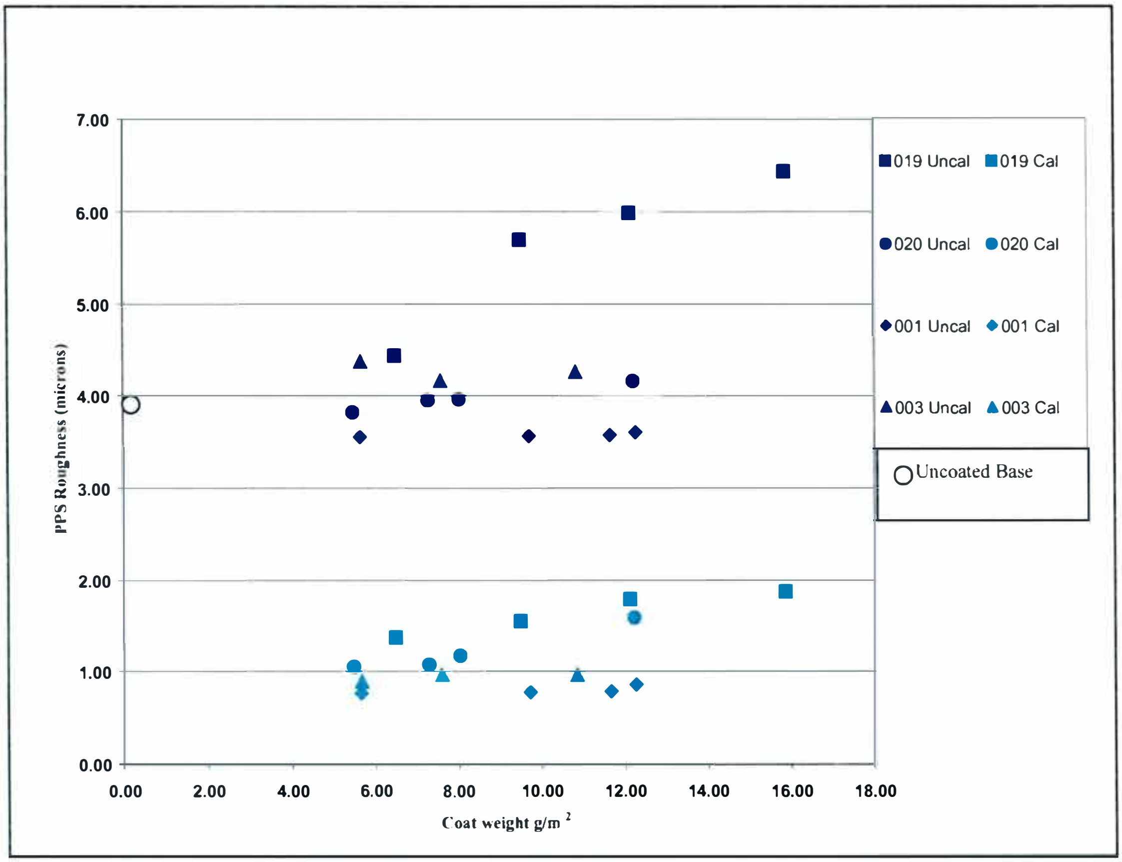


Figure 10. Influence of Coat Weight on Roughness, Before and After Calendering. All Coatings Were Applied at 26% Solids.

Influence of Coat Weight and Coating Solids on Brightness

Figures 11 – 18 indicate that coat weight had no significant influence on the brightness of calendered and uncalendered samples. However, coating solids did. Brightness increased steadily up to the 26% solids level at which point it began to decrease. Once again, the results indicate that the pigments begin to aggregate above 26% solids. As mentioned earlier, the stability of the coating appeared to be related to pH.

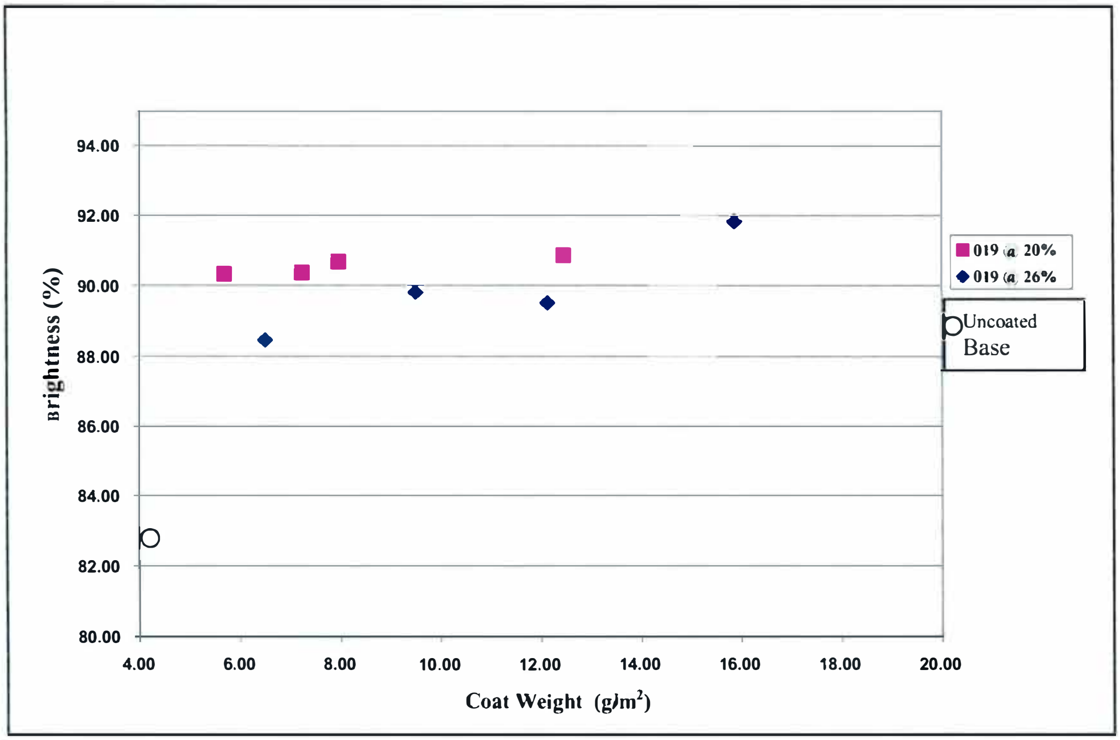


Figure 11. Influence of Coat Weight and Coating Solids on Brightness for the Uncalendered 019 Fumed Silica Coatings.

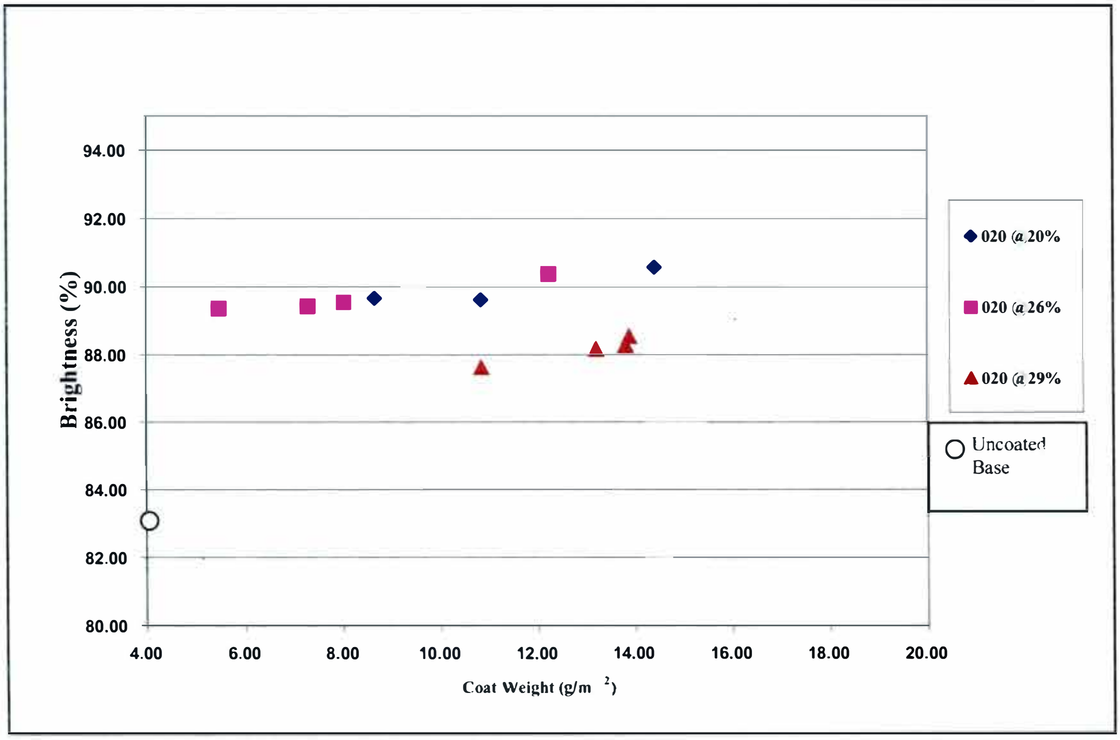


Figure 12. Influence of Coat Weight and Coating Solids on Brightness for the Uncalendered 020 Fumed Silica Coatings.

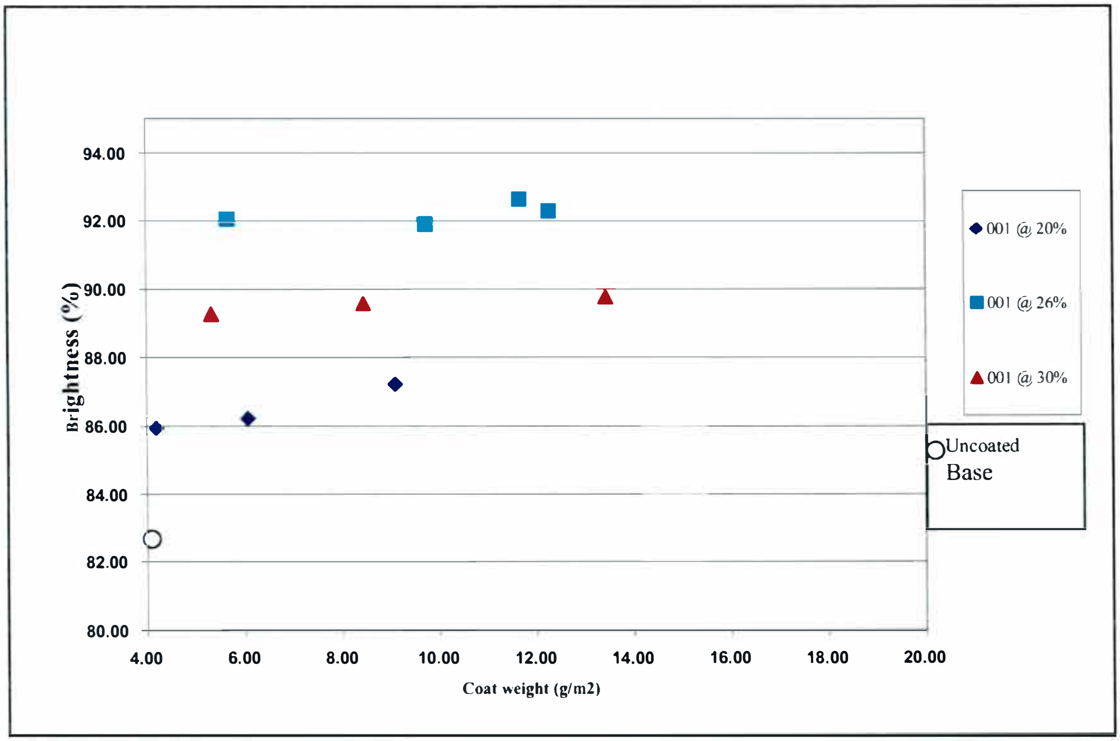


Figure 13. Influence of Coat Weight and Coating Solids on Brightness for the Uncalendered 001 Fumed Silica Coatings.

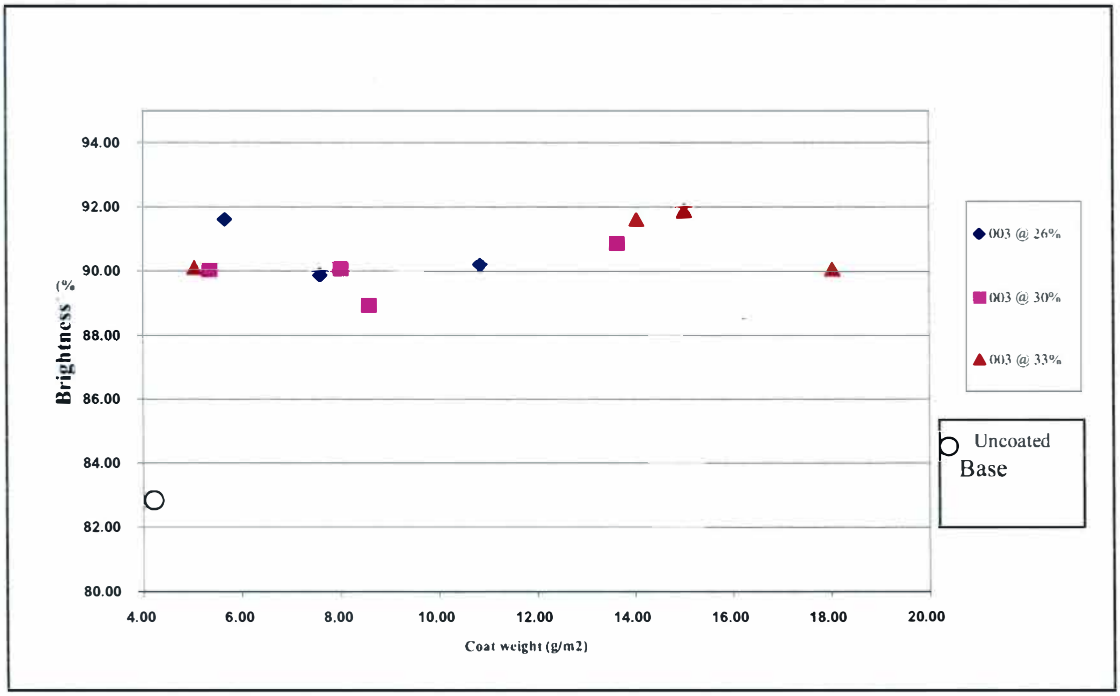


Figure 14. Influence of Coat Weight and Coating Solids on Brightness for the Uncalendered 003 Aluminum Oxide Coatings.

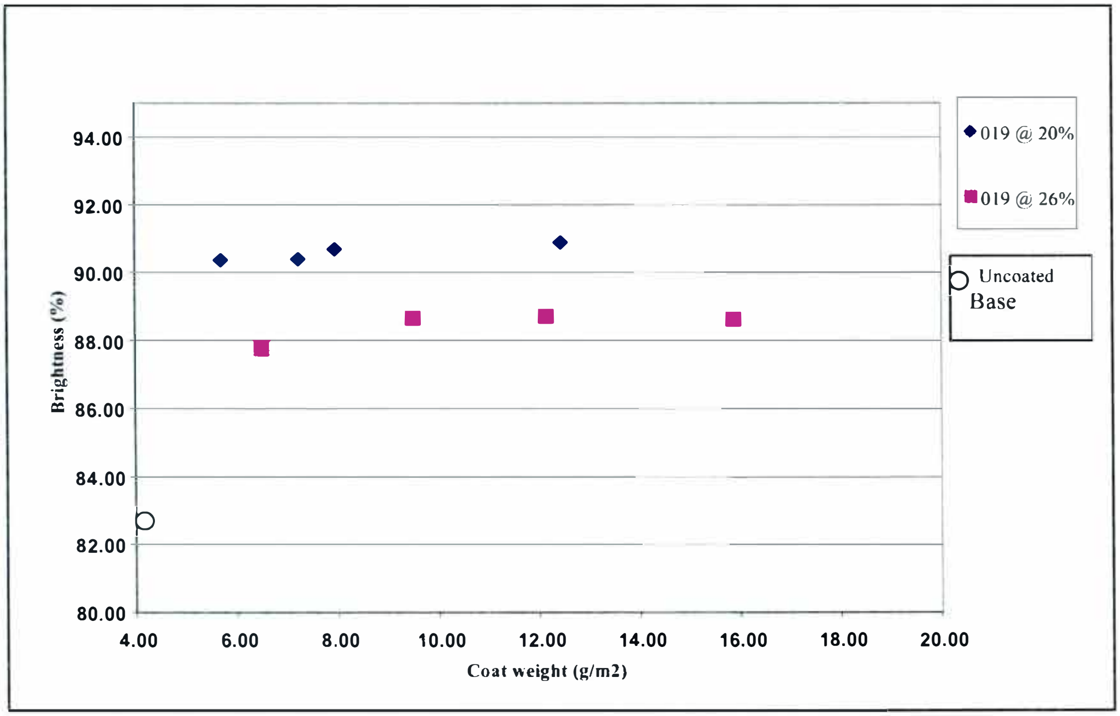


Figure 15. Influence of Coat Weight and Coating Solids on Brightness for the Calendered 019 Fumed Silica Coatings.

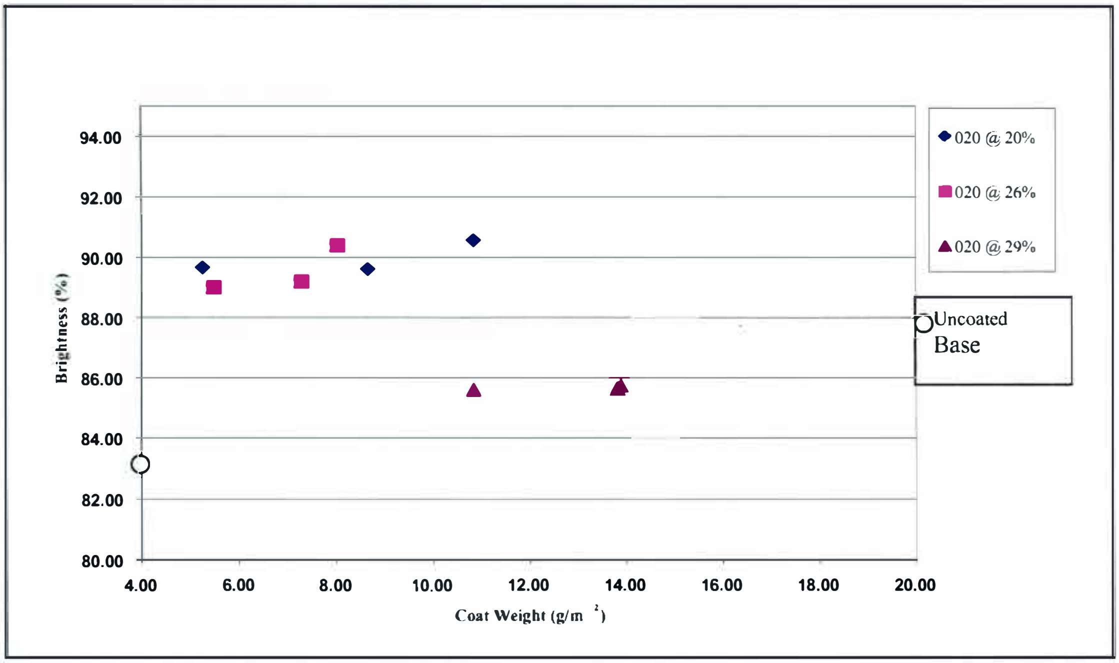


Figure 16. Influence of Coat Weight and Coating Solids on Brightness for the Calendered 020 Fumed Silica Coatings.

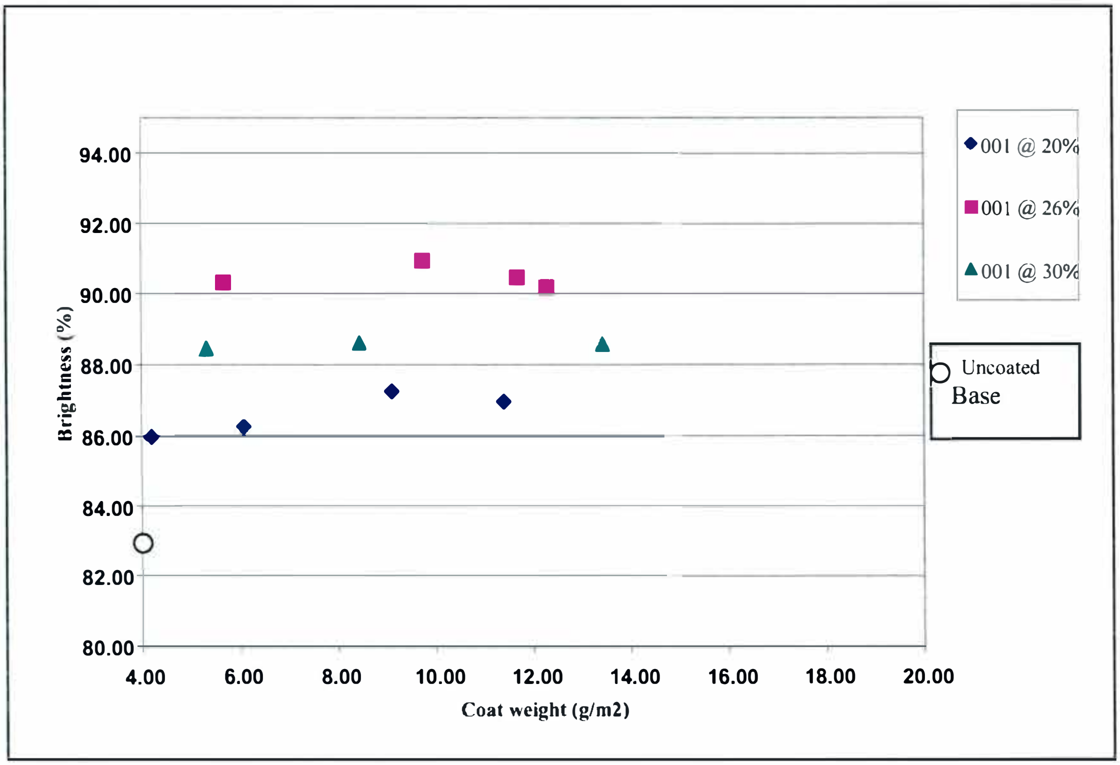


Figure 17. Influence of Coat Weight and Coating Solids on Brightness for the Calendered 001 Fumed Silica Coatings.

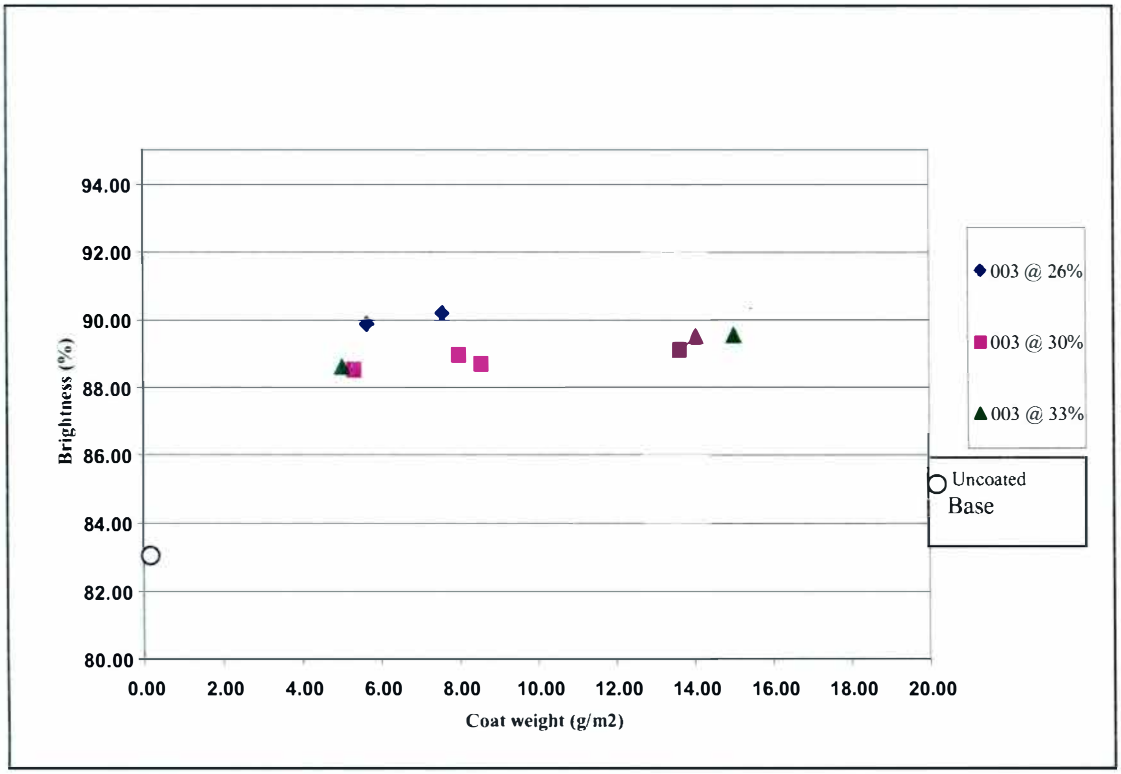


Figure 18. Influence of Coat Weight and Coating Solids on Brightness for the Calendered 003 Aluminum Oxide Coatings.

Calendering had a slight influence on the brightness. As expected, brightness decreased slightly with calendering due to the compaction of the coating layer.

## Influence of Calendering Upon Porosity

Due to the compaction of coating layer or loss in bulk with calendering pressure, the porosity (air permeability) of the coatings decreased with calendering (Figure 19). The mercury intrusion poresimetry data (Figures 56-59, Appendix C) showed all the uncalendered coatings to have a broader pore size distribution in the range of 1 to 7 microns which shifted upon calendering. The pore size distribution of the coatings shifted to right indicating that smaller pores were created by the compaction of larger pores. Regardless of the pigment type used, there was no significant difference in the pore size distribution of the calendered samples (Figure 20).

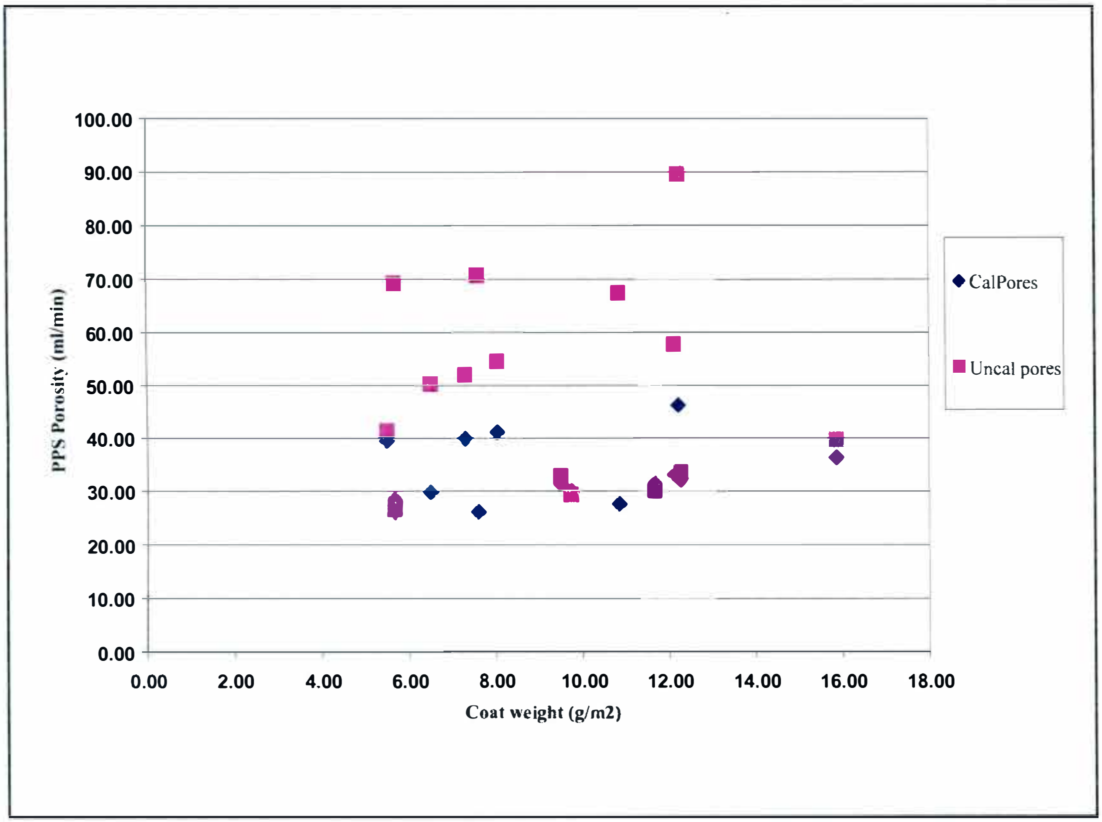


Figure 19. Influence of Calendering on PPS Porosity (1000 KPa) for All Pigments.

Calendering improved the smoothness and gloss of the coatings at all the solids level. Although brightness was not affected significantly by calendering, a significant reduction of porosity was observed. This indicates that although calendering can provide a glossy coating, ink receptivity may be compromised. The effect of calendering on ink hold out was evaluated by measuring the change in contact angle of a liquid drop on the coated surface with time. The test fluid used for these measurements were distilled water. Distilled water was used to simulate the water portion of the inks used in the inkjet printers.

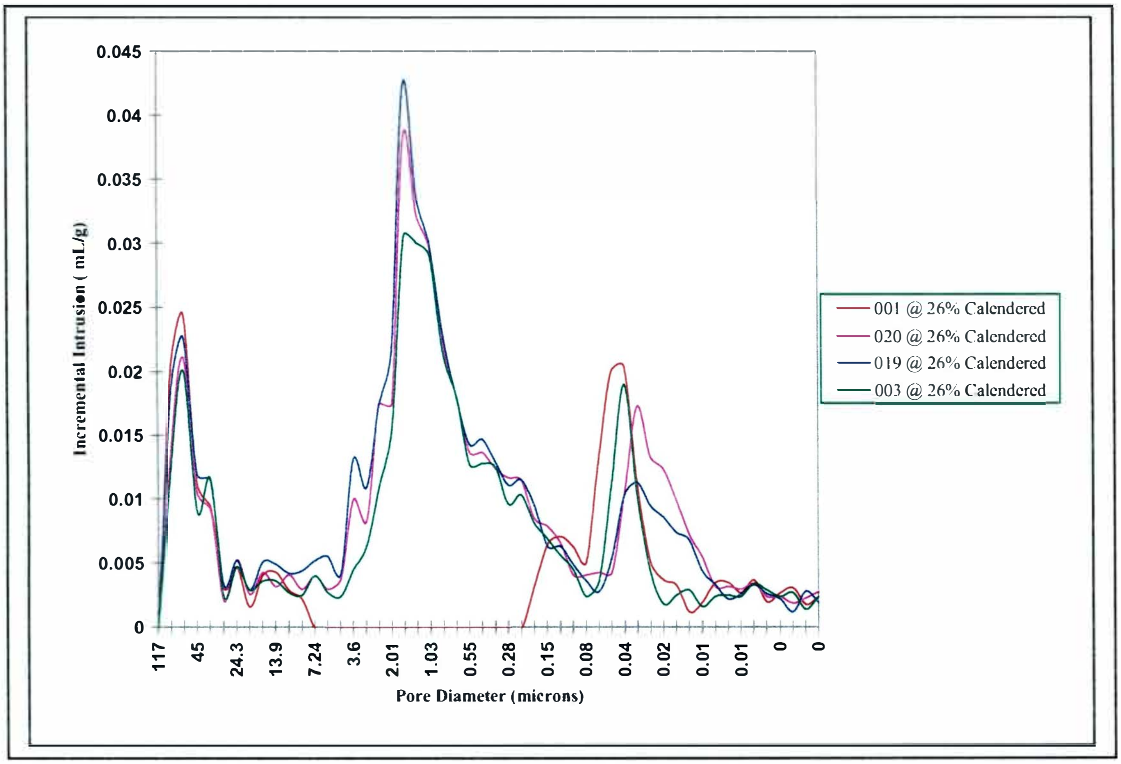


Figure 20. Pore Size Distribution by Mercury Intrusion Porosimetry for Different Coatings at 26% Solids (Calendered).

## Influence of Contact Angle on Ink Density

Figures 21 - 24 show print density to increase with the delta contact angle for the uncalendered samples at 26% solids. Delta contact angle is the difference between the intial and final contact angles within a specified time interval. A high delta contact angle indicates a faster rate of ink absorption. However, the same trend was not observed for the calendered and uncalendered fumed silica coatings at 20% solids.

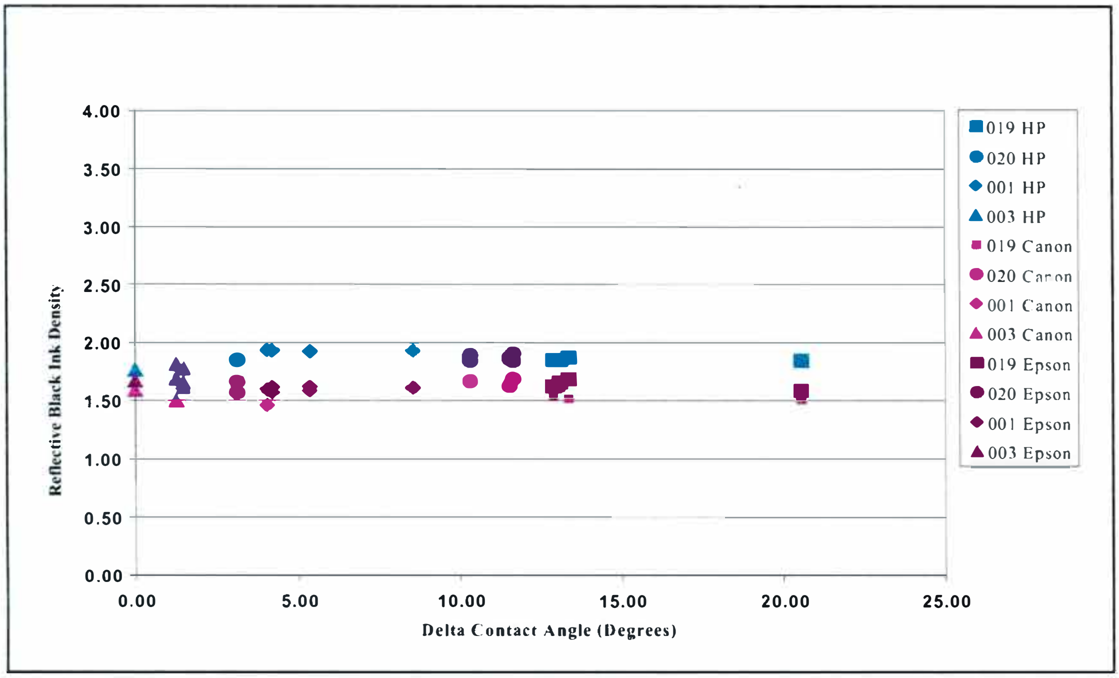


Figure 21. Influence of Contact Angle on Ink Density at 26% Solids (Calendered).

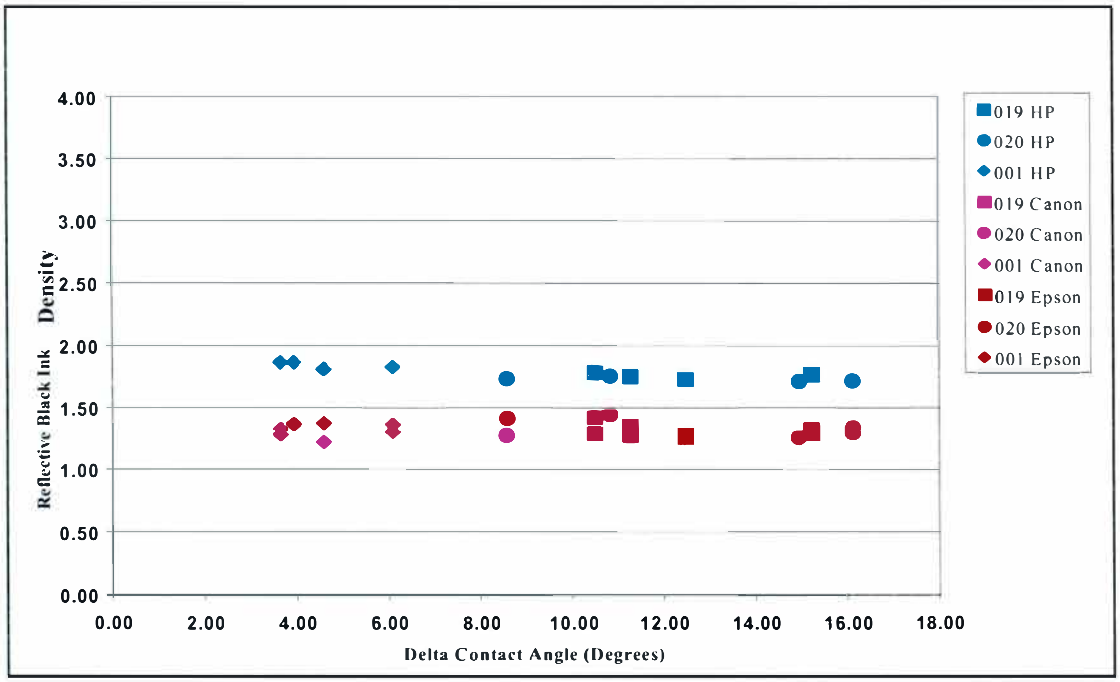


Figure 22. Influence of Contact Angle on Ink Density at 26% Solids (Uncalendered).

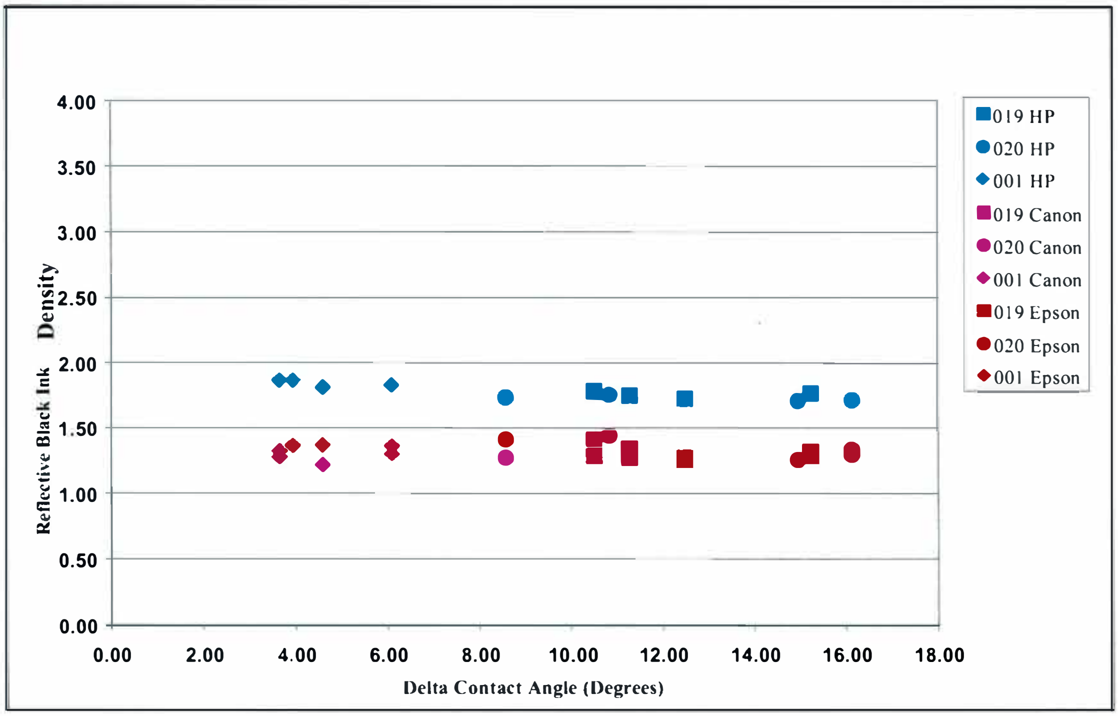


Figure 23. Influence of Contact Angle on Ink Density at 20% Solids (Uncalendered).

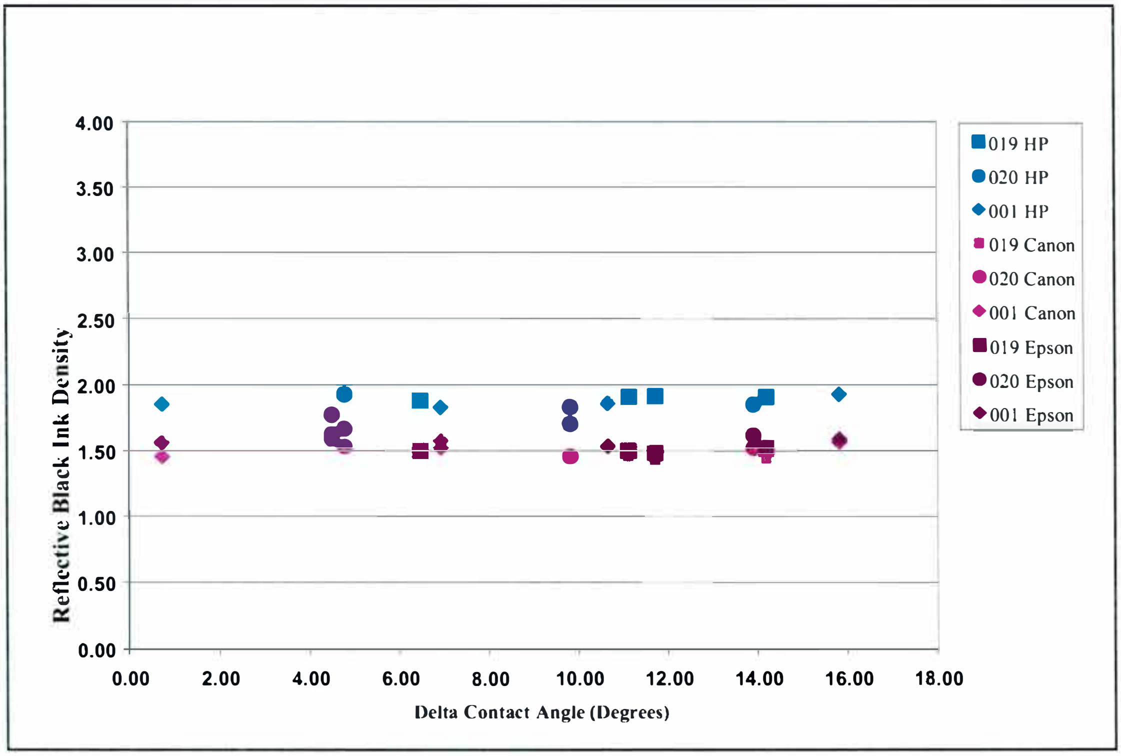


Figure 24. Influence of Contact Angle on Ink Density at 20% Solids (Calendered).

Influence of Roughness on Initial Contact Angle

Figure.25 showed that the initial contact angles were not significantly influenced by the roughness. However, the initial contact angle for fumed silica coatings were significantly different from that of the aluminum oxide coatings.

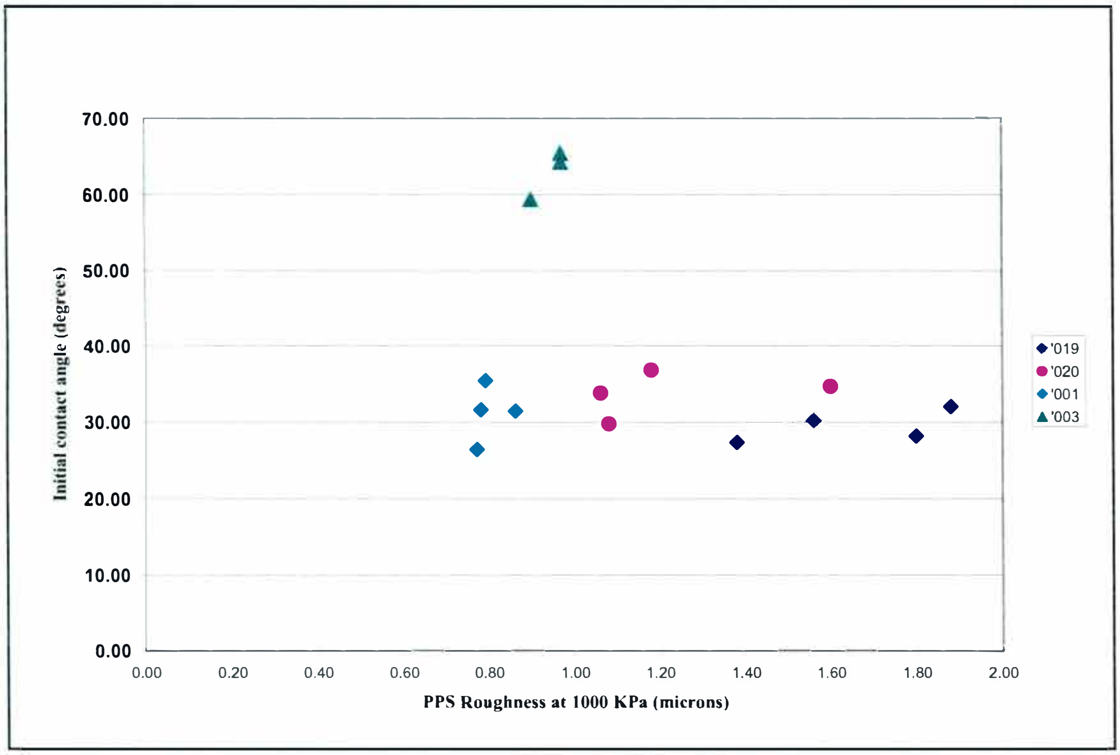


Figure 25. Influence of Roughness on Initial Contact Angle at 20% Solids (Calendered).

Influence of Paper Gloss on Ink Gloss

Increase in paper gloss resulted in higher print gloss on all the fumed silica and aluminum oxide coatings at 26% solids. However, aluminum oxide resulted in lower print gloss compared to 001 type coating. The HP printer resulted in the highest print gloss followed by Epson and Canon respectively. This can be observed from Figures 26 and 27.

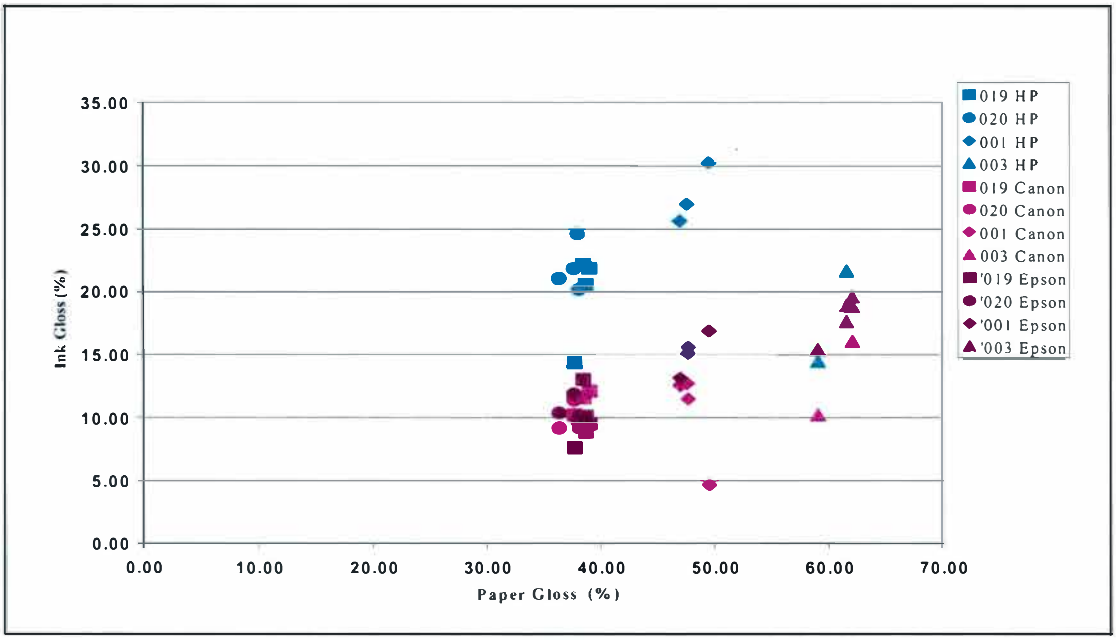


Figure 26. Influence of Paper Gloss on 60° Ink Gloss at 26% Solids (Calendered).

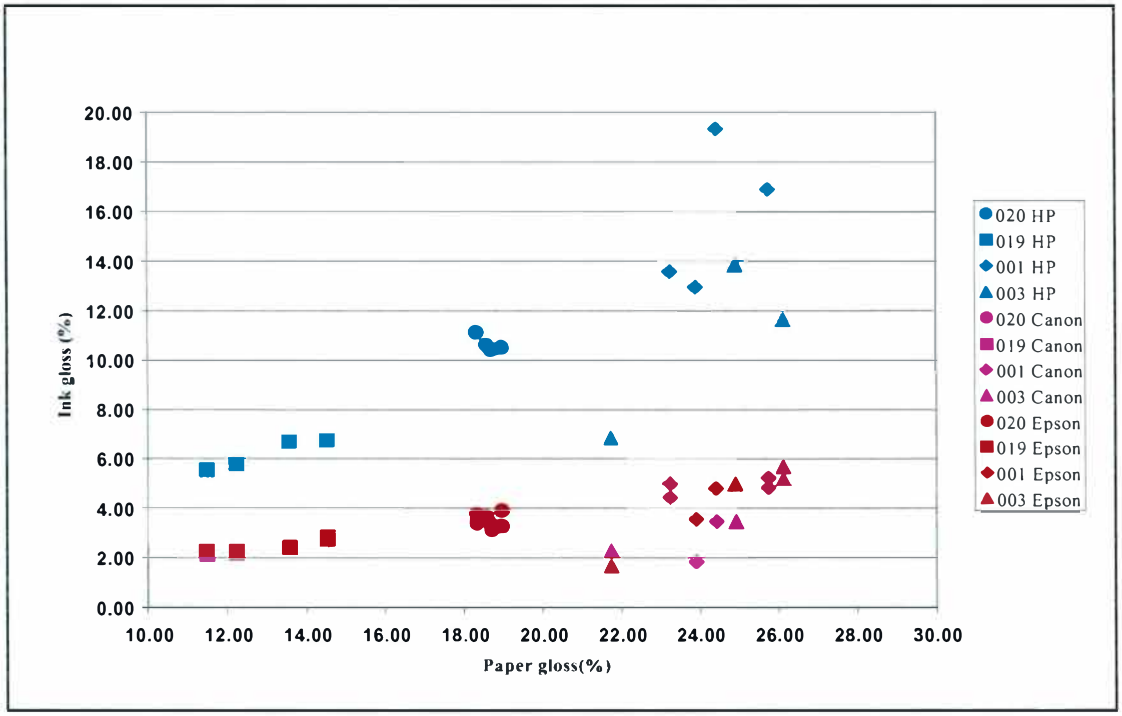


Figure 27. Influence of Paper Gloss on 60° Ink Gloss at 26% Solids (Uncalendered).

Influence of Calendering on Absorption and Spreading

Contact angles increased upon calendering due to the reduction compaction of the coating layer and consequently, the reduction in coating porosity. For all the printers tested, the print densities and dot gains were higher for the calendered samples than the uncalendered samples (Figures 32-55, Appendix B). This is attributed to the higher hold out and spreading of ink on the calendered surfaces due to their reduced porosity properties. The influence of coating porosity on ink density is shown in Figures 28 and 29.

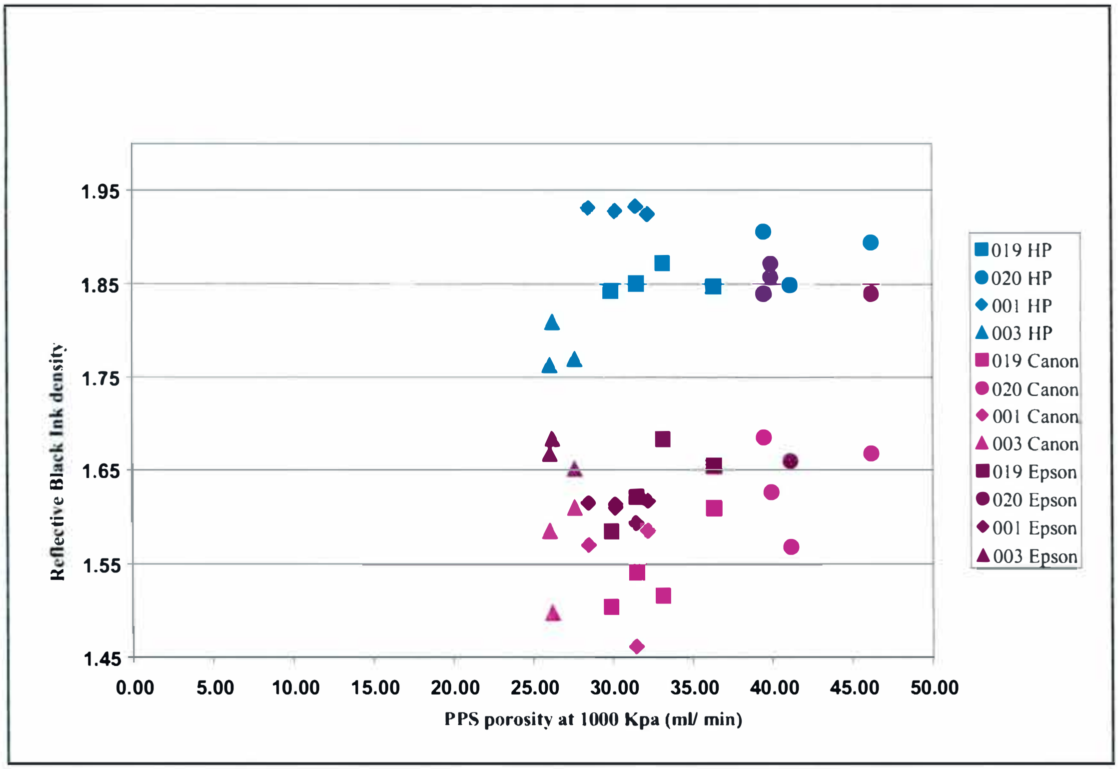


Figure 28. Influence of Paper Porosity on Ink Density at 26% Solids (Calendered).

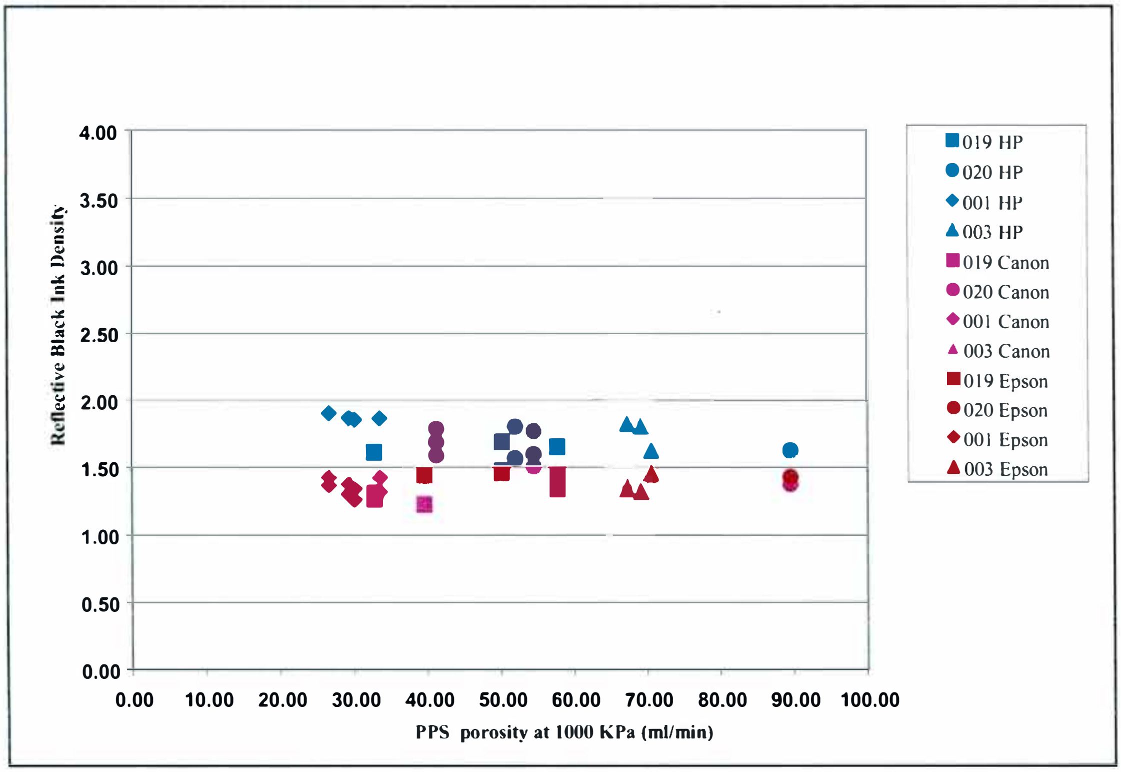


Figure 29. Influence of Paper Porosity on Ink Density at 26% Solids (Uncalendered).

## Influence of Coat Weight on Ink Density

The ink densities of the uncalendered samples increased with coat weight up to 10 $g/m^2$ at which point the ink density did not change. The results indicate coat weights above 10 $g/m^2$ do not provide additional benefits to ink density. Interestingly, calendering had a significant effect on ink density (Figures 30 and 31).

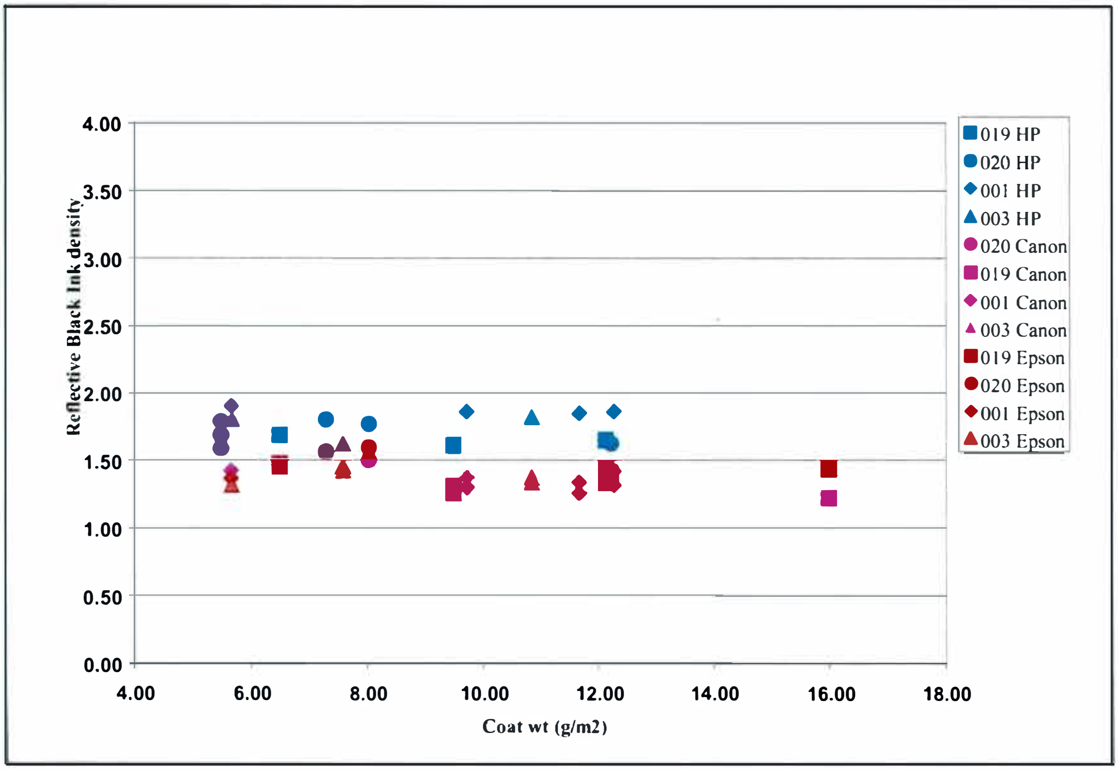


Figure 30. Influence of Coat Weight on Ink density at 26% Solids (Uncalendered ).

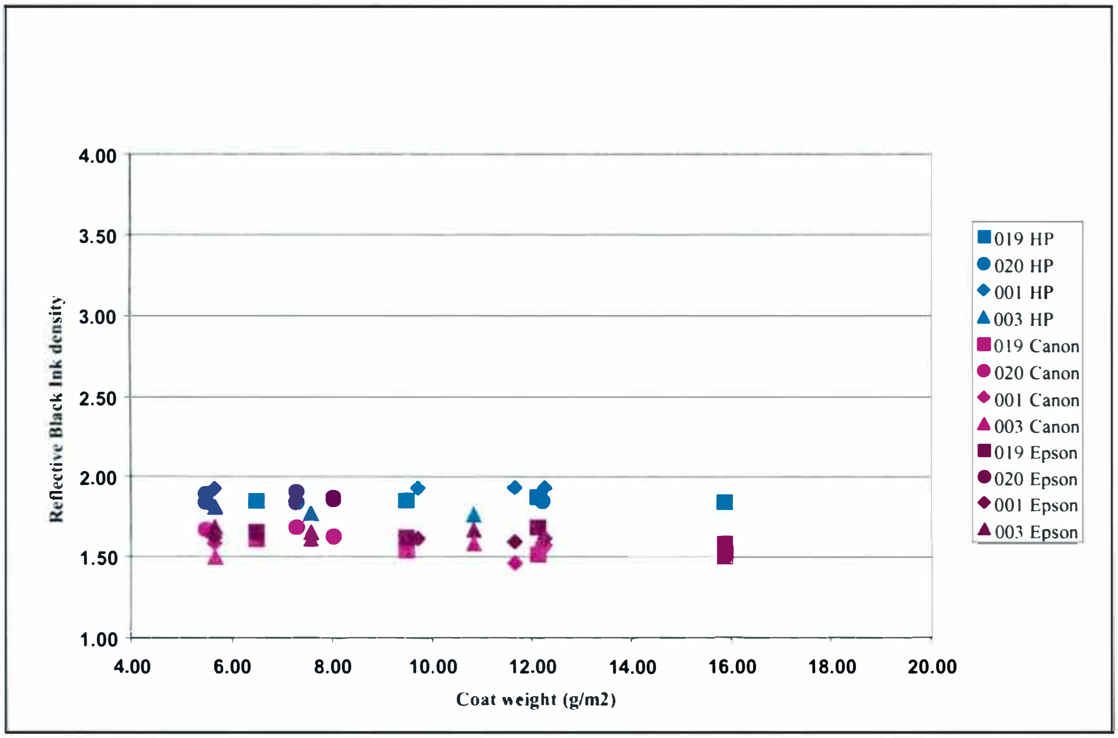


Figure 31. Influence of Coat Weight on Ink Density at 26% Solids (Calendered).

# CHAPTER VI

# CONCLUSIONS

1. The CPVC for the aluminum oxide and the fumed silica pigments occurred at a pigment to binder ratio of 5:1.

2. Calendering was required to obtain gloss values equal to commercial grades inkjet papers.

3. Calendering improved the print density of the aluminum oxide and fumed silica coatings due to decrease in coating porosity.

4. Calendering improved the ink gloss of the aluminum oxide and fumed silica coatings due to increase in paper gloss.

5. The aluminum oxide pigments provided significantly higher gloss values than the silica pigments.

6. Gloss and brightness were not influenced by coat weight.

7. The optical and surface properties were adversely affected when the coatings were prepared at solids levels greater than 26% and 30% for the 019 and 003, respectively.

8. Coatings prepared from the 003 pigment were very sensitive to pH and gelled at pH values greater than 4.5, while the 019 and 020 coatings were sensitive to pH values lower than 10.5.

9. The best print densities and print gloss for all the coated papers were obtained with the HP, Epson and the Canon respectively.

10. CMC thickeners were not compatible with the 003 coatings.

11. The 003 coatings exhibited a higher initial contact angle and lower delta contact angle on calendered samples at 26% solids.

# CHAPTER VII

## RECOMMENDATIONS FOR FUTURE STUDY

Additional studies are required to better understand the influence of pH on the interactivity of the aluminum oxide and silica pigments with PVOH. Studies should include the use of PVOH with a higher degree of hydrolysis in order to obtain better binding strength and/or less binder requirement. More research is also needed to explore the use of synthetic or plastic pigments with the 001 fumed silica to improve its calendered gloss. The incorporation of conventional pigments could also be explored to increase the coating application solids. Higher solid coatings are needed to reduce the basesheet roughening. A high temperature hot/soft nip calendering can be used to enhance the gloss. However, a study of anti-sticking calendering agents may be required.

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APPENDICES

Appendix A

Statistical Analysis (All Data)

Table 4

Classification of Dependent and Independent Variables

| Main effect variables (Independent variables) | Response variables (Dependent variables) |
|---|---|
| Calendering (2) | Brightness |
| Pigment type (4) | Roughness |
| Coat weights (4) | Paper Gloss |
| Solids (4) | Paper Porosity |
| Printer type (3) | Print Density |
| | Print Gloss |
| | Dot Gain |

Table 5

Evaluation of Significant and Most Significant Variable

| Response variable | Main effect variable | F-Value | Pr<F α=0.05 |
|---|---|---|---|
| Brightness | Calendering | 17.69 | 0.0001 |
| | Solids | 8.25 | 0.0001 |
| Roughness | Calendering | 575.06 | 0.0001 |
| | Pigment | 12.26 | 0.0001 |
| | Solids | 4.65 | 0.0057 |
| Paper Gloss | Calendering | 473.33 | 0.0001 |
| | Pigment | 15.35 | 0.0001 |
| | Solids | 3.36 | 0.0251 |
| Porosity | Calendering | 15.5 | 0.0002 |
| | Pigment | 4.2 | 0.0093 |
| Print Density | Pigment | 7.7 | 0.0001 |
| | Solids | 3.32 | 0.0235 |
| | Printer | 136.51 | 0.0001 |
| Dot Gain | Pigment | 5.18 | 0.0024 |
| | Solids | 2.64 | 0.0544 |
| | Printer | 11.1 | 0.0001 |
| Print Gloss | Solids | 3.14 | 0.0294 |
| | Weight | 3.56 | 0.0176 |
| | Printer | 44.67 | 0.0001 |

Table 6

Evaluation of Significant Effects and Interactions

| Response variable | Main effect variable | F-Value | Pr<F α=0.05 |
|---|---|---|---|
| Brightness | Calender | 51.06 | 0.0001 |
| | Pigment | 4.35 | 0.0119 |
| | Solids | 23.81 | 0.0001 |
| | Weight | 0.26 | 0.8560 |
| | Calender*Pigment | 2.80 | 0.0577 |
| | Pigment*Solids | 23.82 | 0.0001 |
| | Calender*Solids | 0.12 | 0.9468 |
| | Calender*Weight | 2.87 | 0.0535 |
| | Solids*Weight | 0.69 | 0.6760 |
| | Pigment*Weight | 2.04 | 0.0920 |
| Roughness | Calender | 1175.26 | 0.0001 |
| | Pigment | 25.05 | 0.0001 |
| | Solids | 9.50 | 0.0002 |
| | Weight | 3.60 | 0.0253 |
| | Calender*Pigment | 10.38 | 0.0001 |
| | Pigment*Solids | 9.36 | 0.0001 |
| | Calender*Solids | 1.39 | 0.2654 |
| | Calender*Weight | 1.09 | 0.3682 |
| | Solids*Weight | 0.51 | 0.8171 |
| | Pigment*Weight | 0.63 | 0.7047 |
| Paper Gloss | Calender | 952.08 | 0.0001 |
| | Pigment | 30.88 | 0.0001 |
| | Solids | 6.76 | 0.0014 |
| | Weight | 0.81 | 0.4979 |
| | Calender*Pigment | 11.74 | 0.0001 |
| | Pigment*Solids | 3.96 | 0.0111 |
| | Calender*Solids | 1.65 | 0.1993 |
| | Calender*Weight | 0.35 | 0.7913 |
| | Solids*Weight | 1.61 | 0.1731 |
| | Pigment*Weight | 2.22 | 0.0691 |

Table 6- continued

| Response variable | Main effect variable | F-Value | Pr<F α=0.05 |
|---|---|---|---|
| Porosity | Calender | 23.29 | 0.0001 |
| | Pigment | 6.31 | 0.0011 |
| | Solids | 0.98 | 0.4120 |
| | Calender*Pigment | 5.05 | 0.0040 |
| | Pigment*Solids | 4.58 | 0.0032 |
| | Calender*Solids | 1.88 | 0.1452 |
| Print Density | Pigment | 19.07 | 0.0001 |
| | Solids | 8.23 | 0.0001 |
| | Weight | 0.20 | 0.8943 |
| | Printer | 338.29 | 0.0001 |
| | Pigment*Solids | 14.59 | 0.0001 |
| | Solids*Printer | 4.66 | 0.0005 |
| | Pigment*Printer | 9.71 | 0.0001 |
| Dot Gain | Pigment | 10.31 | 0.0001 |
| | Solids | 5.26 | 0.0025 |
| | Weight | 0.76 | 0.5199 |
| | Printer | 22.09 | 0.0001 |
| | Pigment*Solid | 10.56 | 0.0001 |
| | Solids*Printer | 4.40 | 0.0008 |
| | Pigment*Printer | 5.58 | 0.0001 |
| Print Gloss | Pigment | 1.74 | 0.1673 |
| | Weight | 3.28 | 0.0262 |
| | Solids | 4.53 | 0.0059 |
| | Printer | 52.09 | 0.0001 |
| | Solids*Weight | 1.65 | 0.1374 |
| | Solids*Printer | 1.41 | 0.2249 |
| | Weight*Printer | 2.25 | 0.0489 |

Table 7

Evaluation of Significant Main Effects – by Calendering and Calendering/Pigment Type

| Response variable | Main effect variable | F-Value | Pr<F α=0.05 |
|---|---|---|---|
| | Uncalendered | | |
| Brightness | Pigment | 2.34 | 0.1003 |
| | Solids | 5.97 | 0.0037 |
| | Weight | 1.16 | 0.3481 |
| | Uncalendered/Pigment A | | |
| Brightness | Solids | 10.10 | 0.0864 |
| | Weight | 4.82 | 0.1766 |
| | Uncalendered/Pigment B | | |
| Brightness | Solids | 3.94 | 0.1447 |
| | Weight | 0.63 | 0.6427 |
| | Uncalendered/Pigment C | | |
| Brightness | Solids | 16.90 | 0.0112 |
| | Weight | 2.08 | 0.2404 |
| | Uncalendered/Pigment D | | |
| Brightness | Solids | 9.62 | 0.0496 |
| | Weight | 2.45 | 0.2342 |
| | Calendered | | |
| Brightness | Pigment | 0.61 | 0.6153 |
| | Solids | 2.92 | 0.0558 |
| | Weight | 0.20 | 0.8929 |

Table 7- continued

| Response variable | Main effect variable | F-Value | Pr<F α=0.05 |
|---|---|---|---|
| | Calendered/Pigment A | | |
| Brightness | Solids | 206.08 | 0.0048 |
| | Weight | 5.41 | 0.1600 |
| | Calendered/Pigment B | | |
| Brightness | Solids | 27.19 | 0.0120 |
| | Weight | 1.18 | 0.4476 |
| | Calendered/Pigment C | | |
| Brightness | Solids | 128.01 | 0.0002 |
| | Weight | 3.38 | 0.1380 |
| | Calendered/Pigment D | | |
| Brightness | Solids | 4.27 | 0.1325 |
| | Weight | 0.35 | 0.7314 |
| | Uncalendered | | |
| Roughness | Pigment | 11.76 | 0.0001 |
| | Solids | 2.42 | 0.0921 |
| | Weight | 1.48 | 0.2474 |
| | Uncalendered/Pigment A | | |
| Roughness | Solids | 9.41 | 0.0919 |
| | Weight | 1.23 | 0.4782 |
| | Uncalendered/Pigment B | | |
| Roughness | Solids | 13.40 | 0.0319 |
| | Weight | 3.90 | 0.1464 |

Table 7- continued

| Response variable | Main effect variable | F-Value | Pr<F α=0.05 |
|---|---|---|---|
| | Uncalendered/Pigment C | | |
| Roughness | Solids | 691.81 | 0.0001 |
| | Weight | 1.03 | 0.4345 |
| | Uncalendered/Pigment D | | |
| Roughness | Solids | 1.44 | 0.3635 |
| | Weight | 0.72 | 0.5542 |
| | Calendered | | |
| Roughness | Pigment | 5.37 | 0.0060 |
| | Solids | 11.07 | 0.0001 |
| | Weight | 1.34 | 0.2859 |
| | Calendered/Pigment A | | |
| Roughness | Solids | 26.41 | 0.0358 |
| | Weight | 1.83 | 0.3719 |
| | Calendered/Pigment B | | |
| Roughness | Solids | 1.81 | 0.3055 |
| | Weight | 1.70 | 0.3360 |
| | Calendered/Pigment C | | |
| Roughness | Solids | 37.81 | 0.0025 |
| | Weight | 0.92 | 0.4699 |

Table 7- continued

| Response variable | Main effect variable | F-Value | Pr<F α=0.05 |
|---|---|---|---|
| | Calendered/Pigment D | | |
| Roughness | Solids | 4.42 | 0.1276 |
| | Weight | 2.56 | 0.2247 |
| | Uncalendered | | |
| Paper Gloss | Pigment | 3.55 | 0.0301 |
| | Solids | 4.69 | 0.0107 |
| | Weight | 0.75 | 0.5329 |
| | Uncalendered/Pigment A | | |
| Paper Gloss | Solids | 20.74 | 0.0450 |
| | Weight | 1.08 | 0.5128 |
| | Uncalendered/Pigment B | | |
| Paper Gloss | Solids | 0.23 | 0.8041 |
| | Weight | 1.36 | 0.4039 |
| | Uncalendered/Pigment C | | |
| Paper Gloss | Solids | 45.71 | 0.0018 |
| | Weight | 2.01 | 0.2484 |

Table 7- continued

| Response variable | Main effect variable | F-Value | Pr<F α=0.05 |
|---|---|---|---|
| | Uncalendered/Pigment D | | |
| Paper Gloss | Solids | 12.23 | 0.0361 |
| | Weight | 0.38 | 0.7140 |
| | Calendered | | |
| Paper Gloss | Pigment | 18.10 | 0.0001 |
| | Solids | 2.07 | 0.1319 |
| | Weight | 0.25 | 0.8636 |
| | Calendered/Pigment A | | |
| Paper Gloss | Solids | 1.59 | 0.3344 |
| | Weight | 0.17 | 0.9065 |
| | Calendered/Pigment B | | |
| Paper Gloss | Solids | 0.63 | 0.5890 |
| | Weight | 0.94 | 0.5207 |
| | Calendered/Pigment C | | |
| Paper Gloss | Solids | 50.05 | 0.0015 |
| | Weight | 1.30 | 0.3677 |
| | Calendered/Pigment D | | |
| Paper Gloss | Solids | 3.02 | 0.1913 |
| | Weight | 3.92 | 0.1456 |
| | Uncalendered | | |
| Porosity | Pigment | 4.53 | 0.0111 |
| | Solids | 1.09 | 0.3710 |

Table 7- continued

| Response variable | Main effect variable | F-Value | Pr<F α=0.05 |
|---|---|---|---|
| | Uncalendered/Pigment A | | |
| Porosity | Solids | 4.52 | 0.0869 |
| | Uncalendered/Pigment B | | |
| Porosity | Solids | 6.01 | 0.0369 |
| | Uncalendered/Pigment C | | |
| Porosity | Solids | 590.31 | 0.0001 |
| | Uncalendered/Pigment D | | |
| Porosity | Solids | 43.44 | 0.0007 |
| | Calendered | | |
| Porosity | Pigment | 1.17 | 0.3389 |
| | Solids | 2.53 | 0.0795 |
| | Calendered/Pigment A | | |
| Porosity | Solids | 0.31 | 0.6019 |
| | Calendered/Pigment B | | |
| Porosity | Solids | 15.32 | 0.0044 |
| | Calendered/Pigment C | | |
| Porosity | Solids | 79.48 | 0.0001 |
| | Calendered/Pigment D | | |
| Porosity | Solids | 0.85 | 0.4819 |

Table 8

Evaluation of Significant Effects for Phase II Statistical Analysis by Printer

| Response variable | Main effect variable | F-Value | Pr<F α=0.05 |
|---|---|---|---|
| | Printer HP | | |
| Print Density | Pigment | 5.07 | 0.0068 |
| | Solids | 0.14 | 0.9348 |
| | Printer Canon | | |
| Print Density | Pigment | 4.32 | 0.0134 |
| | Solids | 3.88 | 0.0205 |
| | Printer Epson | | |
| Print Density | Pigment | 12.70 | 0.0001 |
| | Solids | 2.78 | 0.0609 |
| | Printer HP | | |
| Print Gloss | Weight | 6.51 | 0.0020 |
| | Solids | 3.25 | 0.0377 |
| | Printer Cannon | | |
| Print Gloss | Weight | 0.20 | 0.8985 |
| | Solids | 0.12 | 0.9452 |
| | Printer Epson | | |
| Print Gloss | Weight | 0.49 | 0.6904 |
| | Solids | 0.37 | 0.7755 |

Table 8 - continued

| Response variable | Main effect variable | F-Value | Pr<F α=0.05 |
|---|---|---|---|
| | Printer HP | | |
| Dot Gain | Pigment | 3.32 | 0.0354 |
| | Solids | 8.28 | 0.0005 |
| | Printer Canon | | |
| Dot Gain | Pigment | 3.84 | 0.0212 |
| | Solids | 4.40 | 0.0125 |
| | Printer Epson | | |
| Dot Gain | Pigment | 5.94 | 0.0032 |
| | Solids | 0.66 | 0.5835 |

Appendix B

Influence of Porosity on Ink Density and Dot Gain for All Coatings
With Different Printers

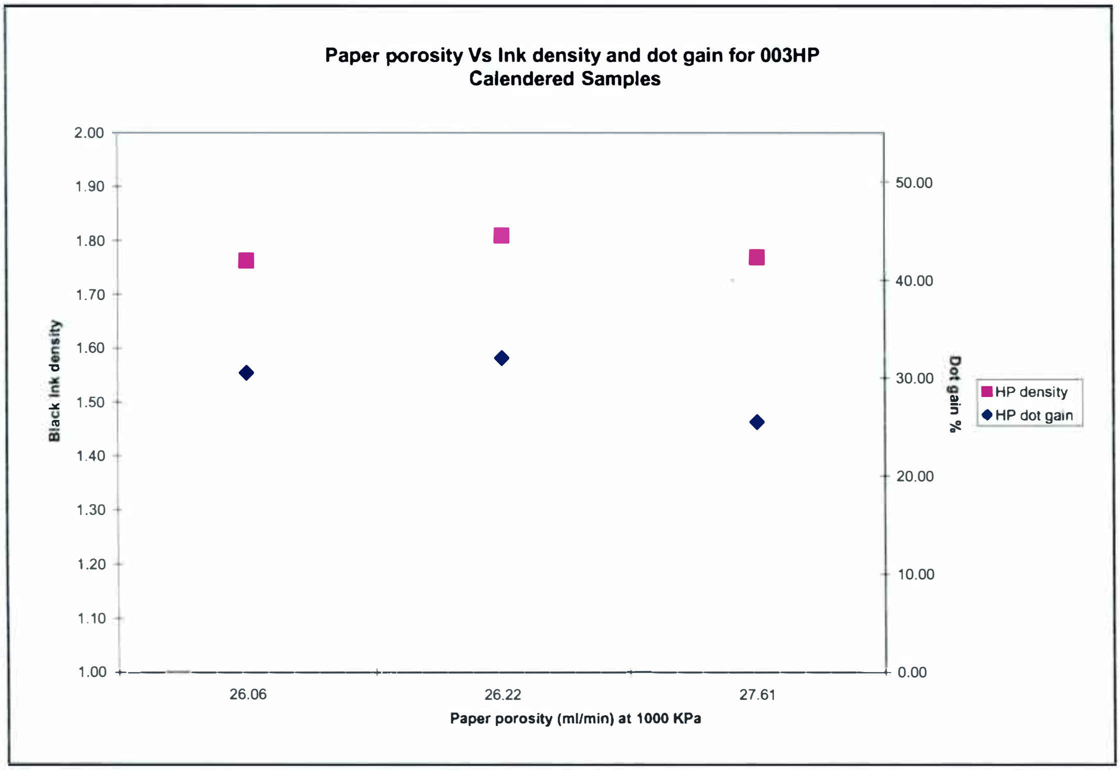


Figure 32. Paper Porosity Vs Ink Density and Dot Gain (003 HP Calendered).

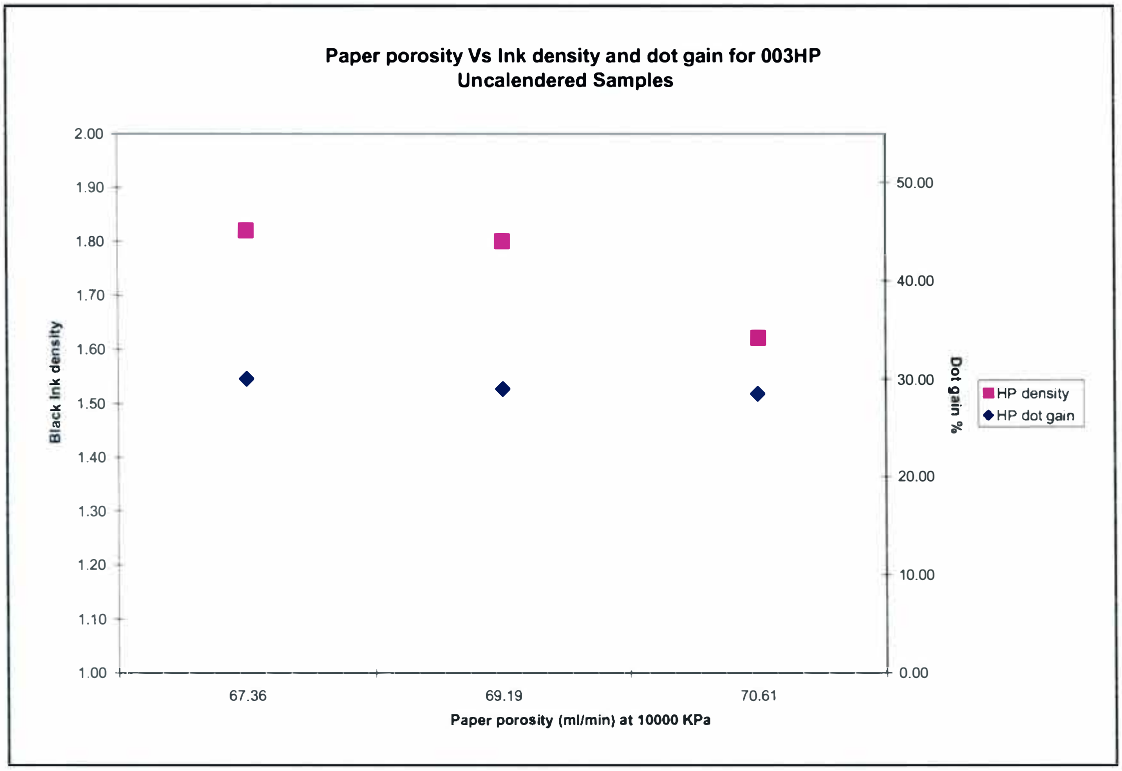


Figure 33. Paper Porosity Vs Ink Density and Dot Gain (003 HP Uncalendered).

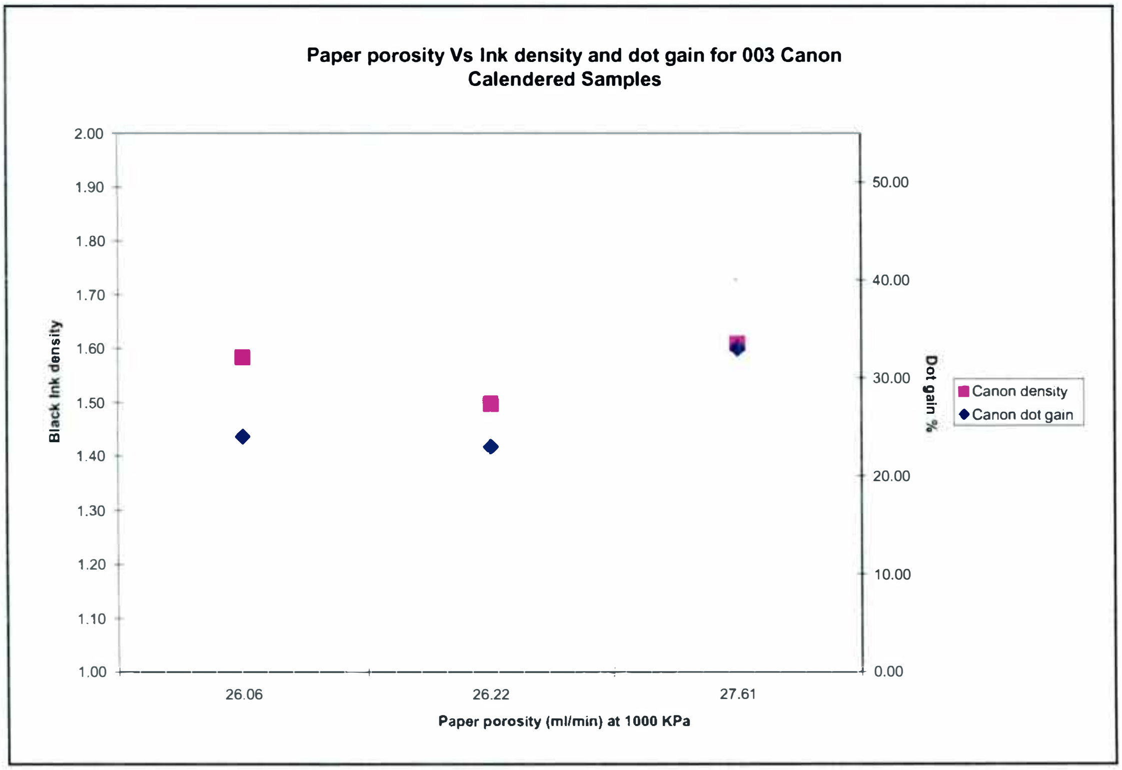


Figure 34. Paper Porosity Vs Ink Density and Dot Gain (003 Canon Calendered).

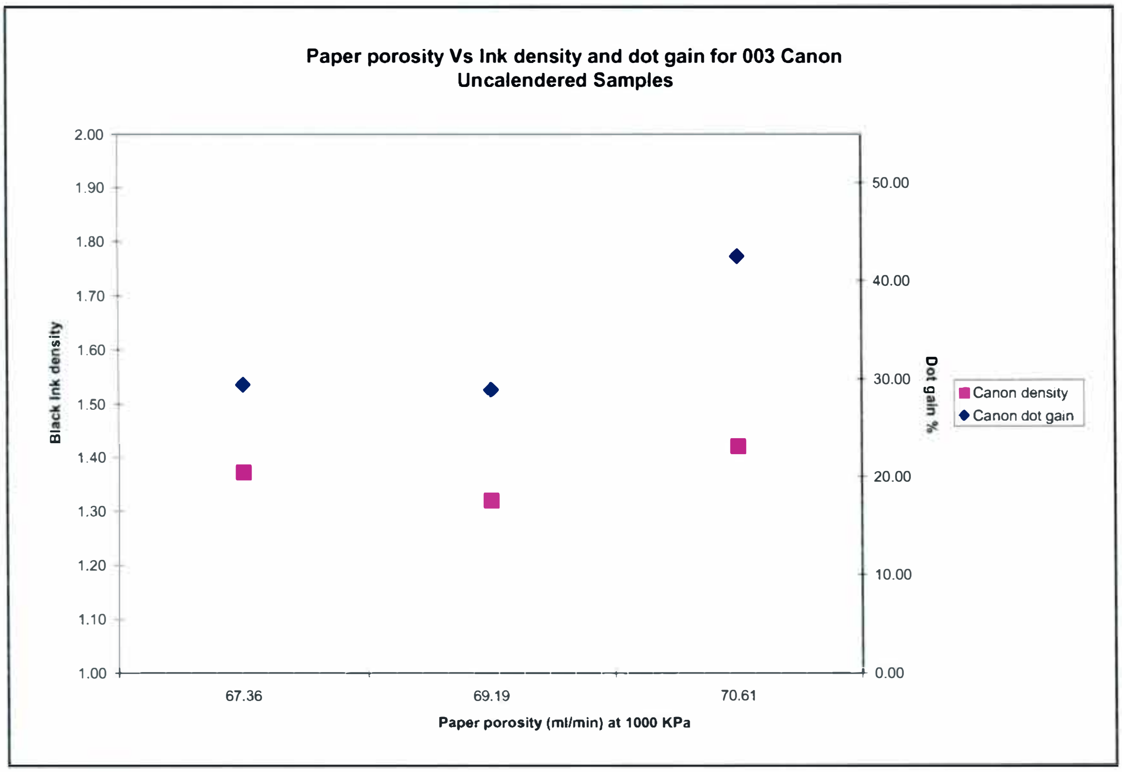


Figure 35. Paper Porosity Vs Ink Density and Dot Gain (003 Canon Uncalendered).

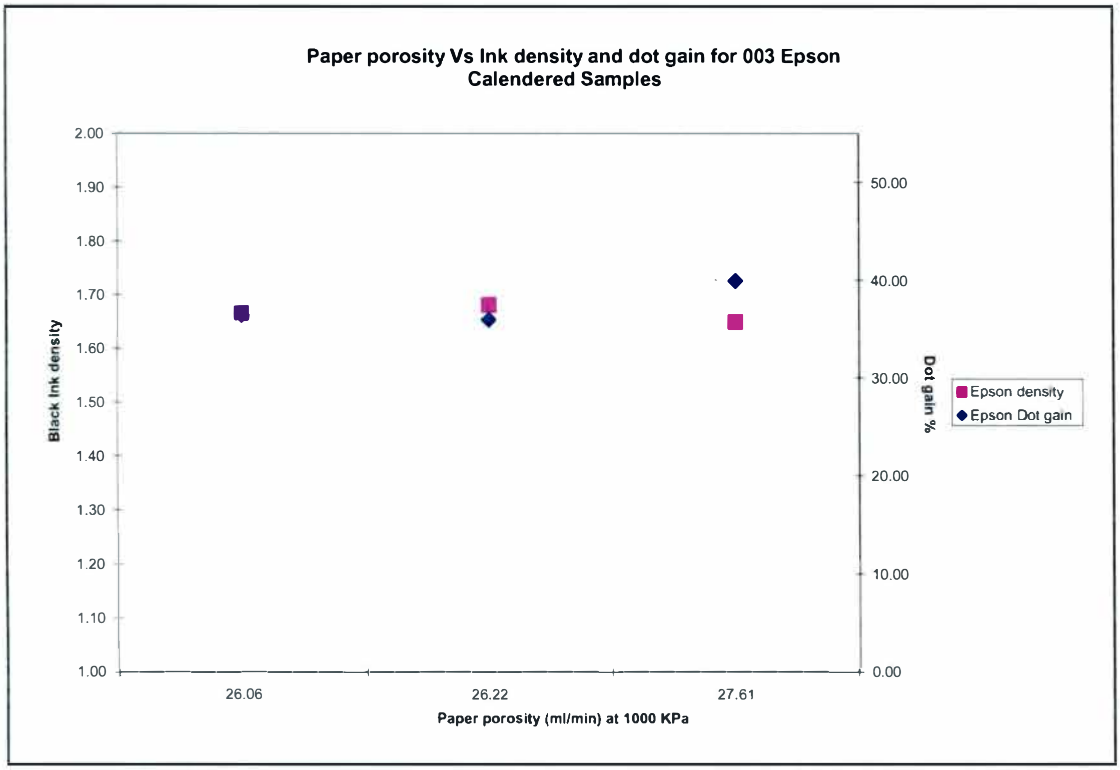


Figure 36. Paper Porosity Vs Ink Density and Dot Gain (003 Epson Calendered).

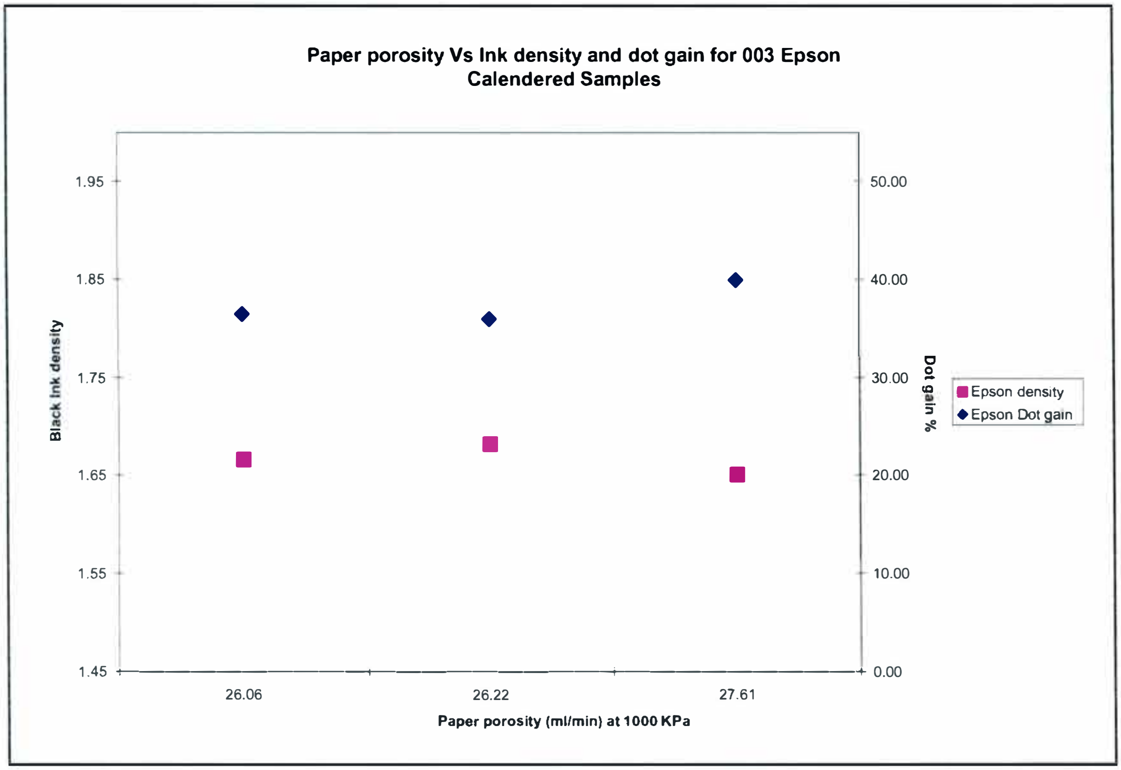


Figure 37. Paper Porosity Vs Ink Density and Dot Gain (003 Epson Uncalendered).

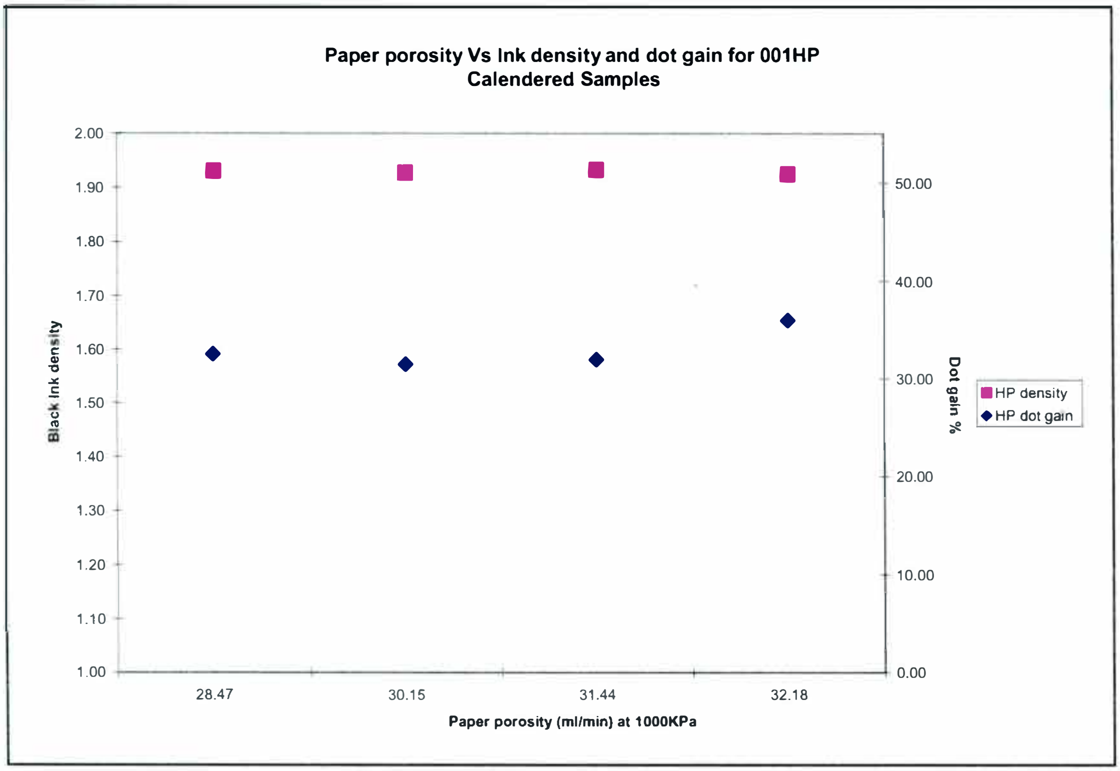


Figure 38. Paper Porosity Vs Ink Density and Dot Gain (001 HP Calendered).

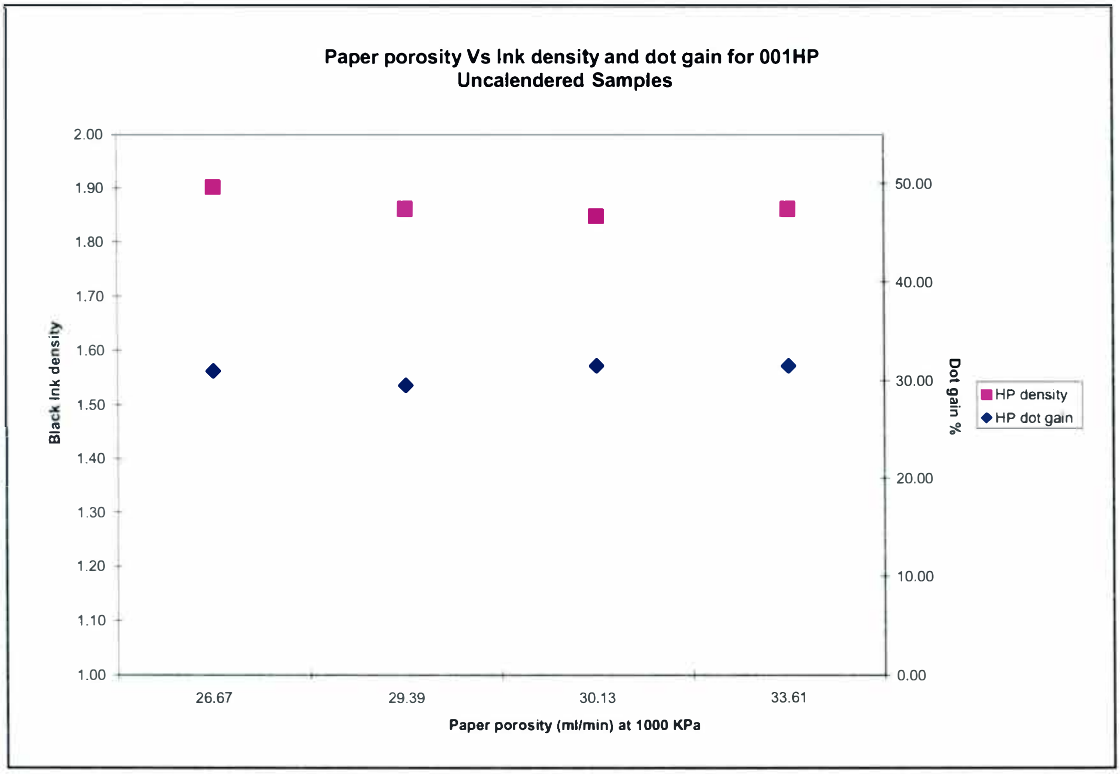


Figure 39. Paper Porosity Vs Ink Density and Dot Gain (001 HP Uncalendered).

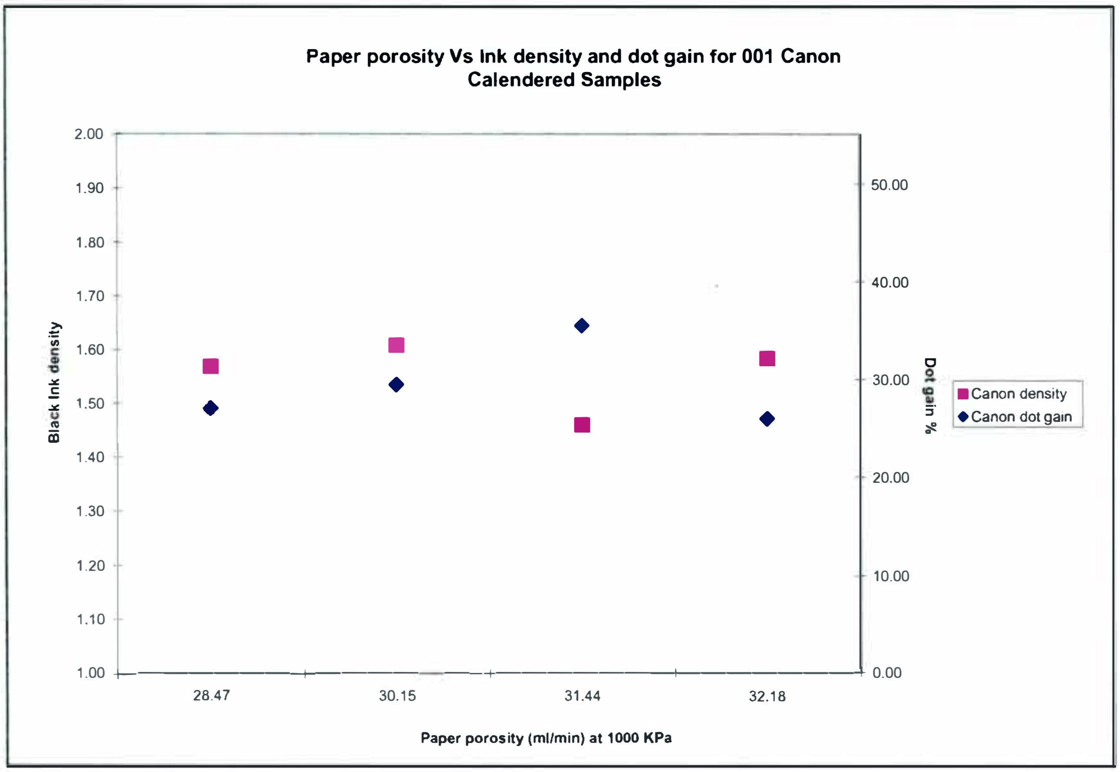


Figure 40. Paper Porosity Vs Ink Density and Dot Gain (001 Canon Calendered).

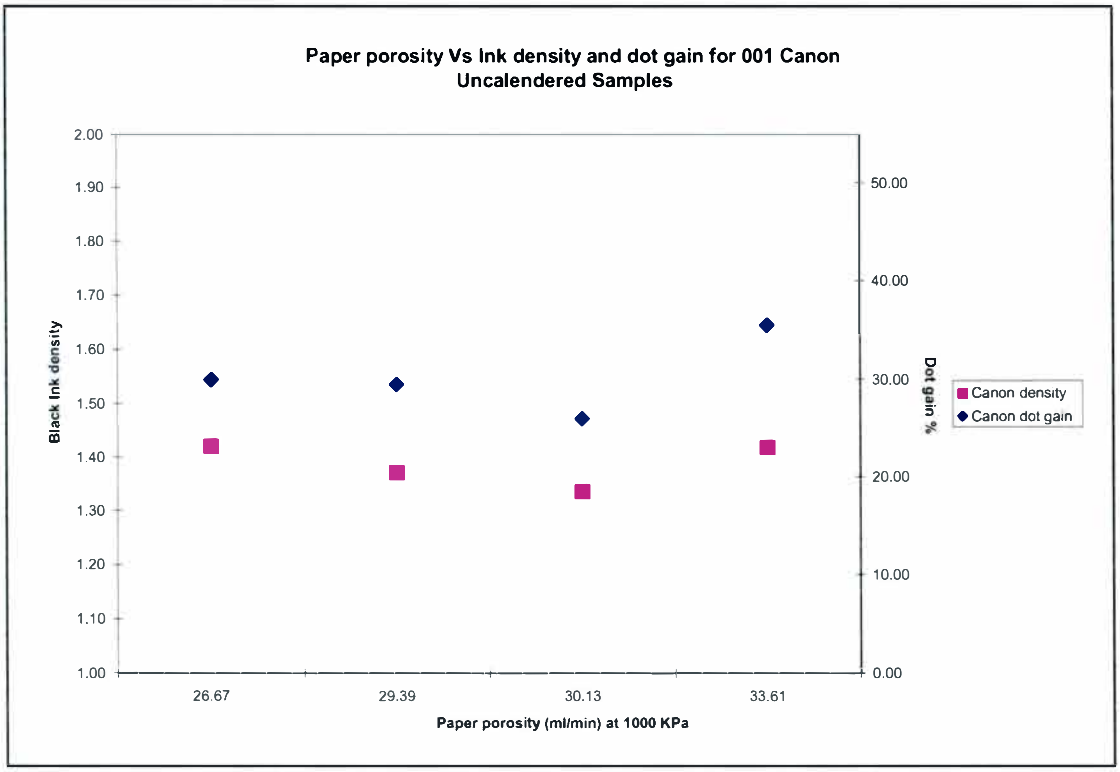


Figure 41. Paper Porosity Vs Ink Density and Dot Gain (001 Canon Uncalendered).

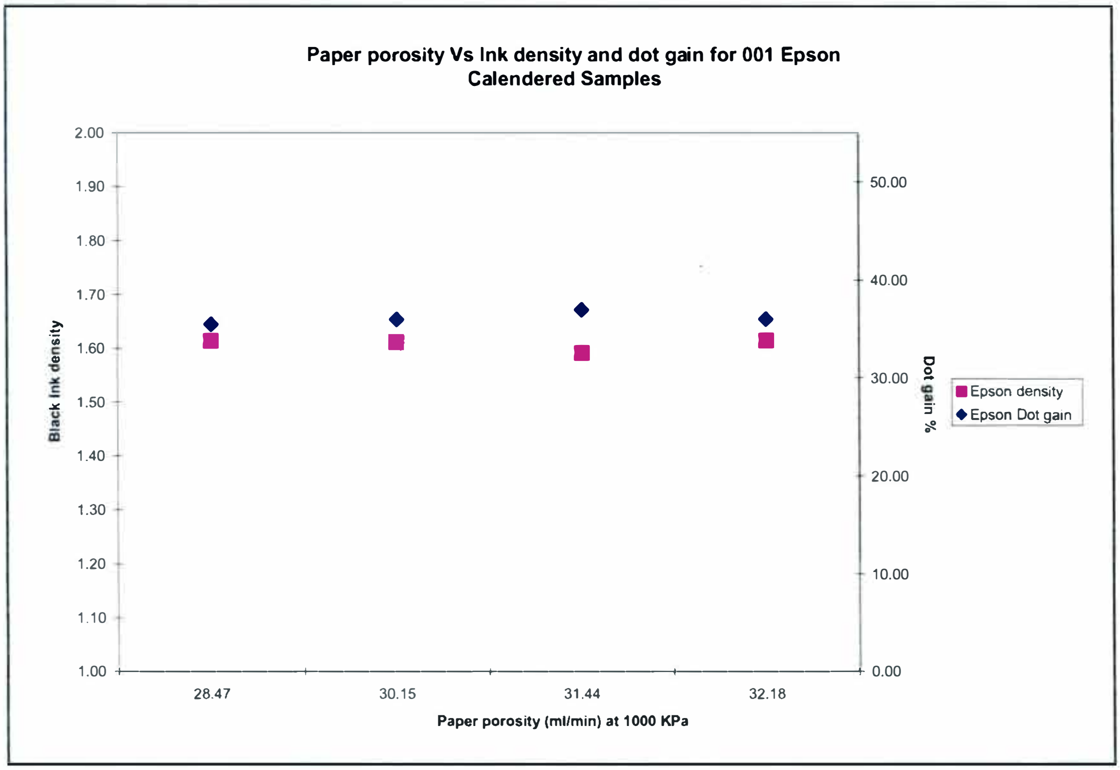


Figure 42. Paper Porosity Vs Ink Density and Dot Gain (001 Epson Calendered).

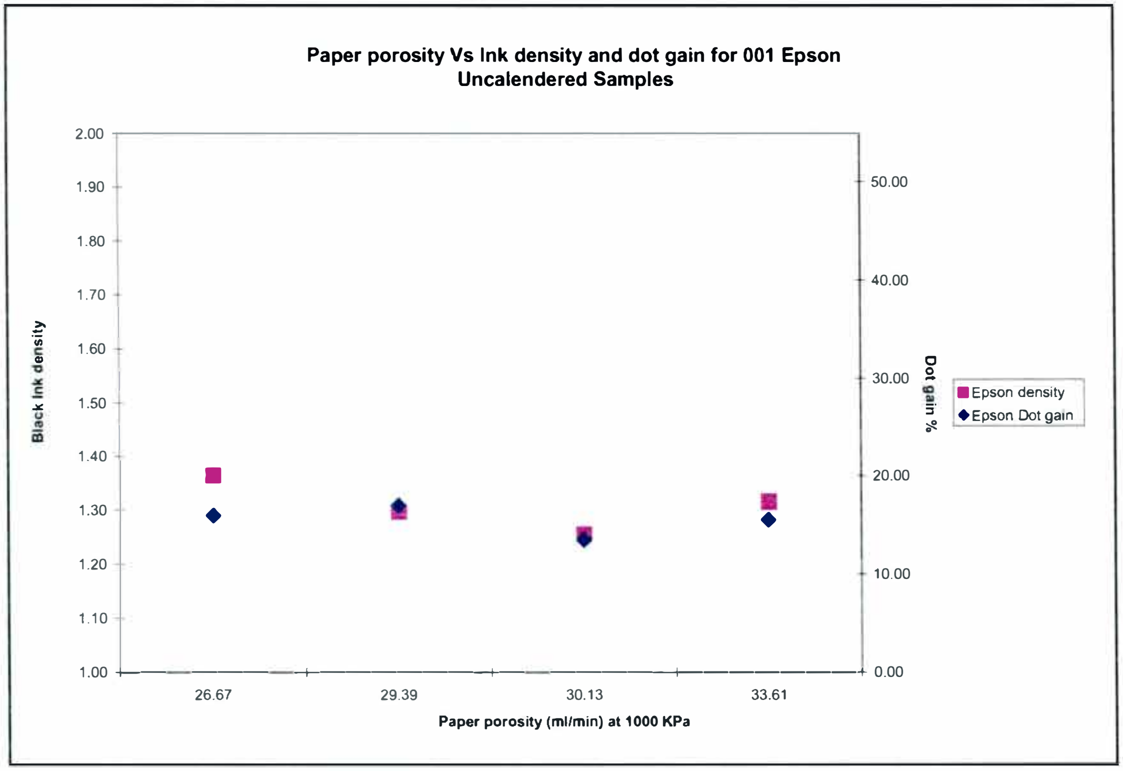


Figure 43. Paper Porosity Vs Ink Density and Dot Gain (001 Epson Uncalendered).

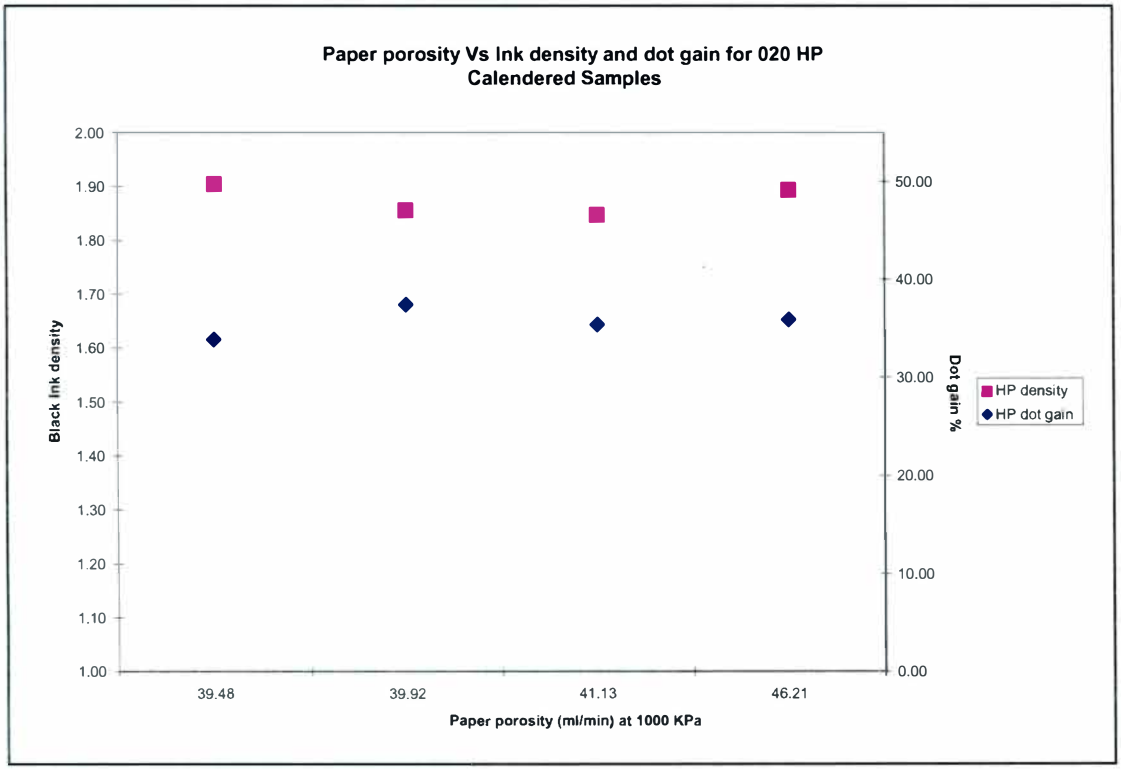


Figure 44. Paper Porosity Vs Ink Density and Dot Gain (020 HP Calendered).

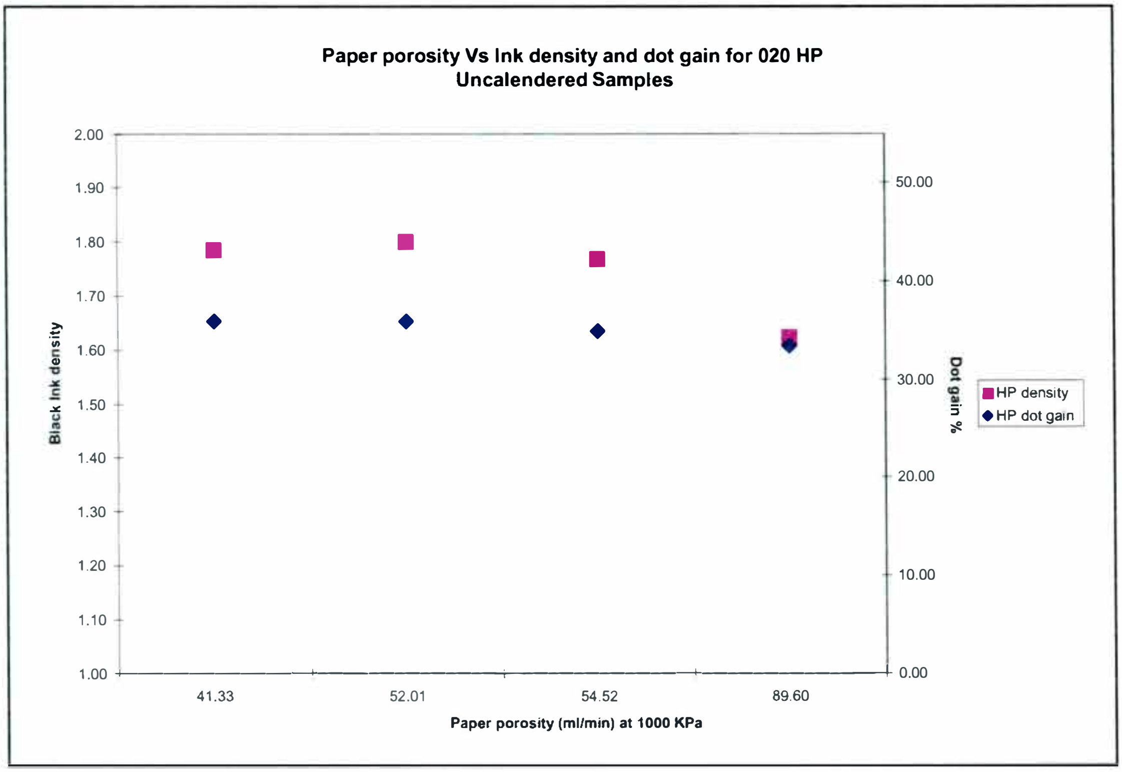


Figure 45. Paper Porosity Vs Ink Density and Dot Gain (020 HP Uncalendered).

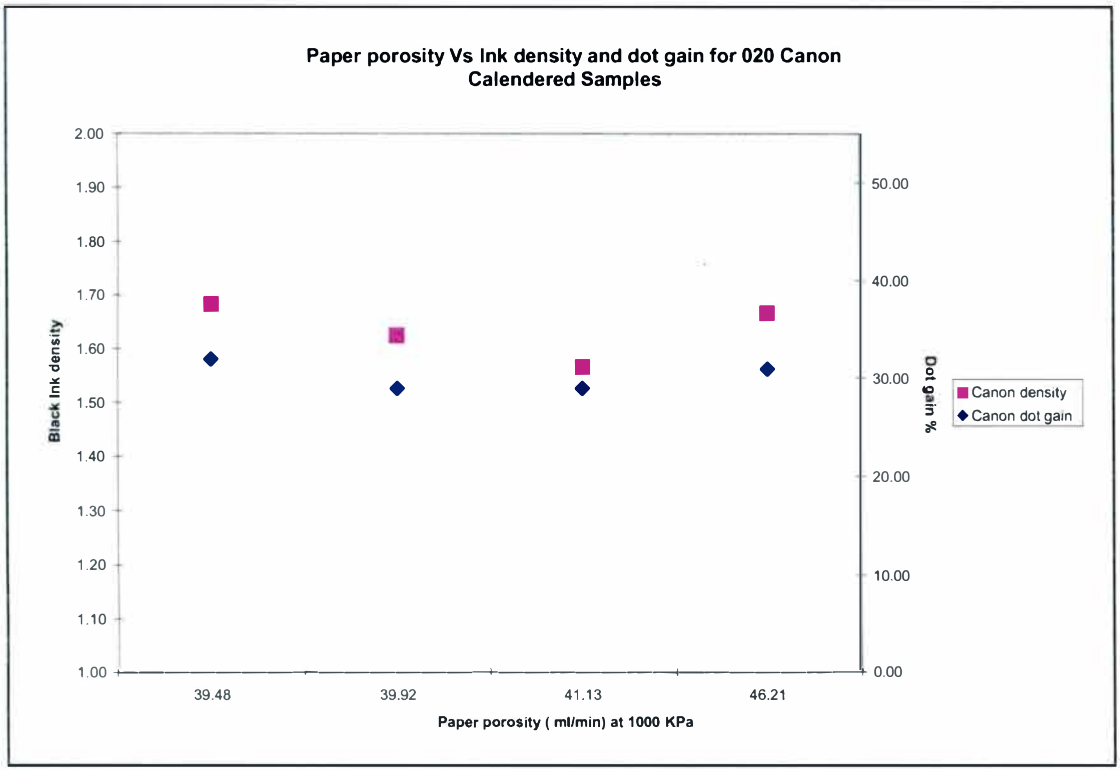


Figure 46. Paper Porosity Vs Ink Density and Dot Gain (020 Canon Calendered).

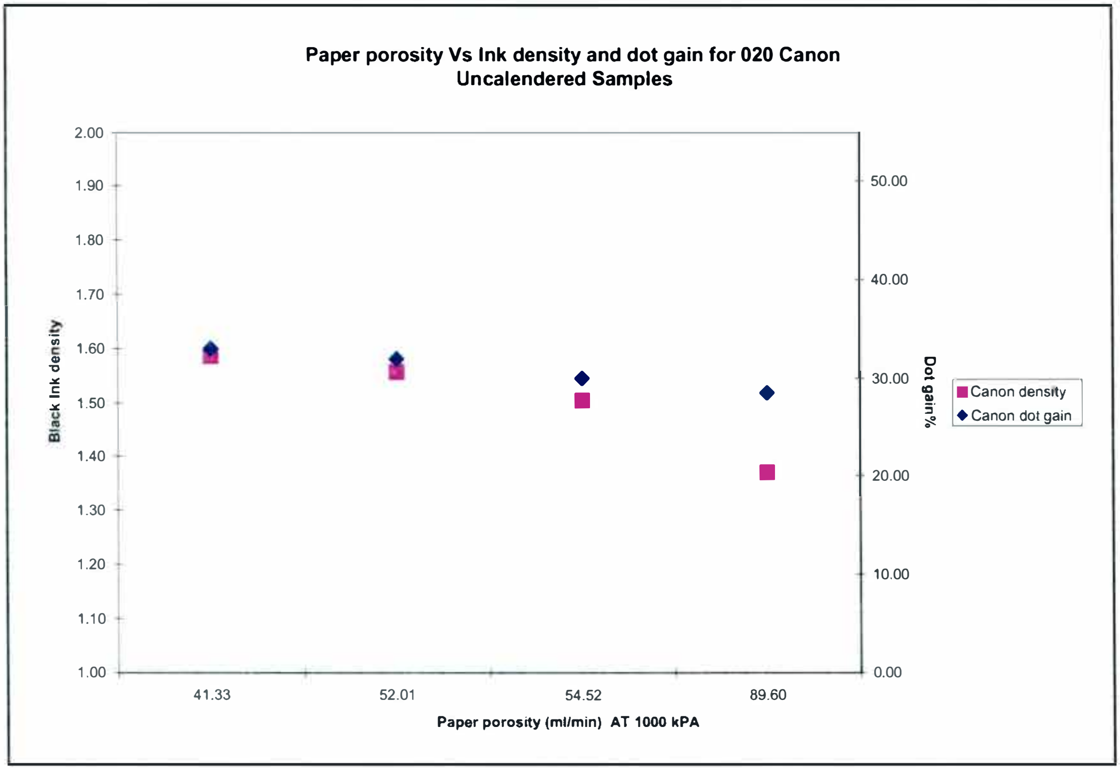


Figure 47. Paper Porosity Vs Ink Density and Dot Gain (020 Canon Uncalendered).

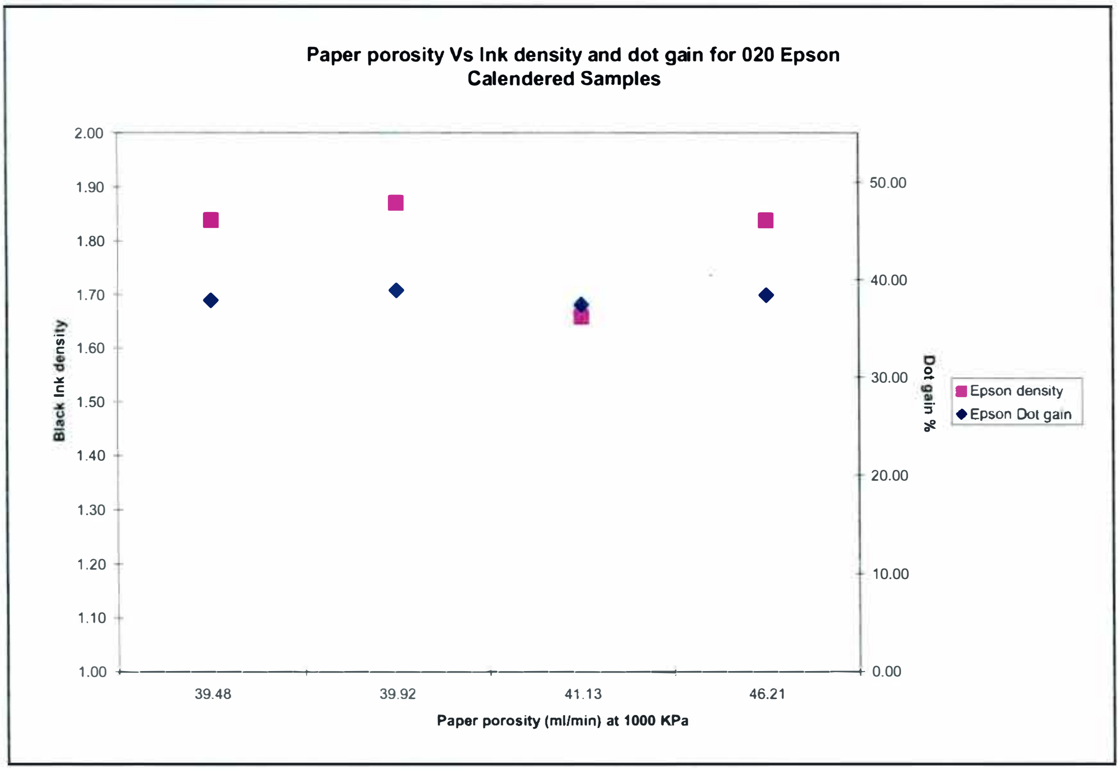


Figure 48. Paper Porosity Vs Ink Density and Dot Gain (020 Epson Calendered).

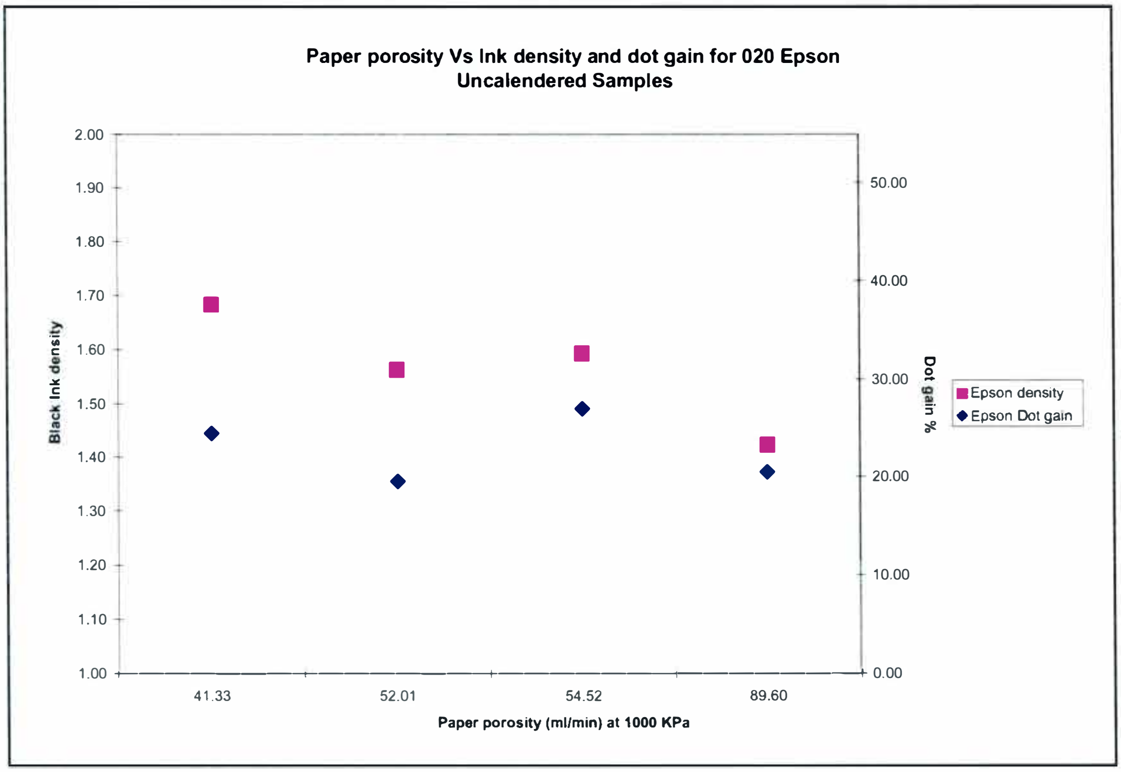


Figure 49. Paper Porosity Vs Ink Density and Dot Gain (020 Epson Uncalendered).

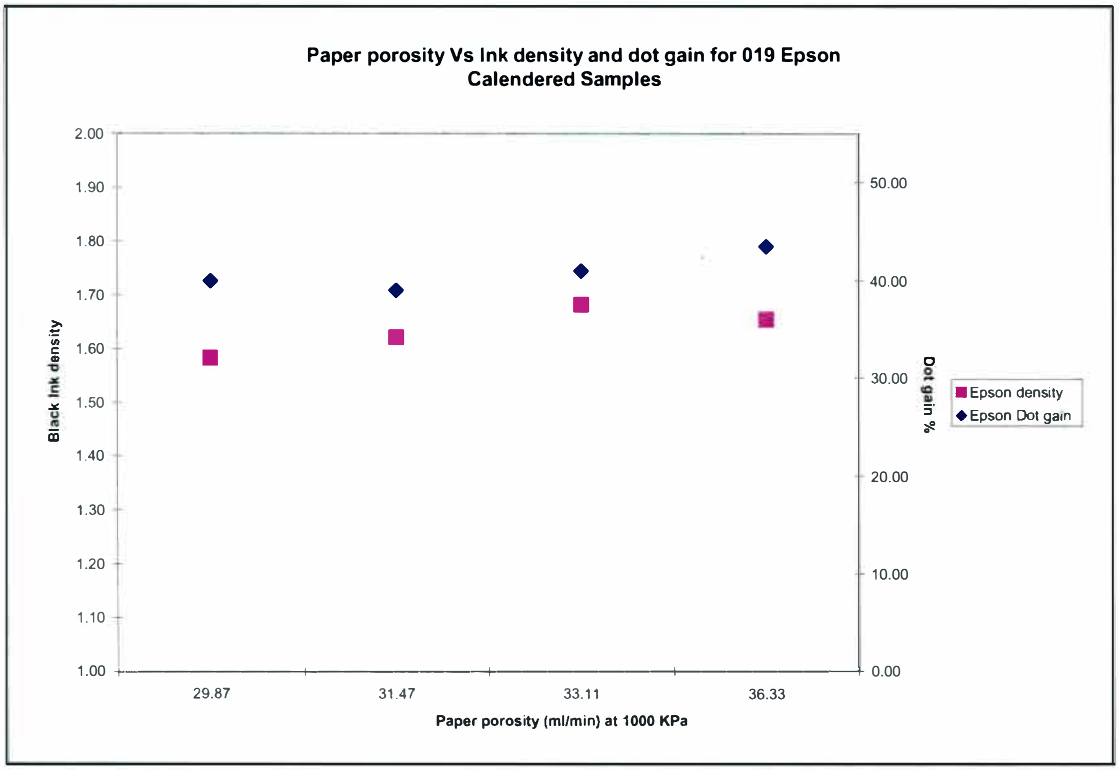


Figure 50. Paper Porosity Vs Ink Density and Dot Gain (019 Epson Calendered).

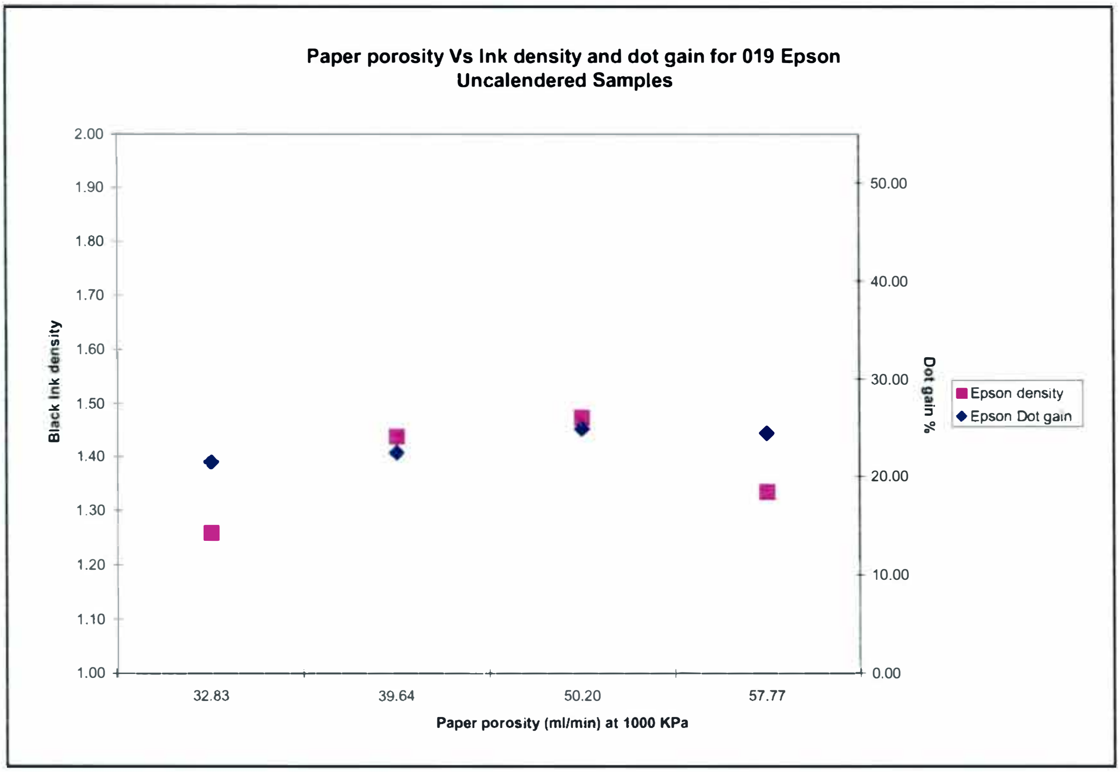


Figure 51. Paper Porosity Vs Ink Density and Dot Gain (019 Epson Uncalendered).

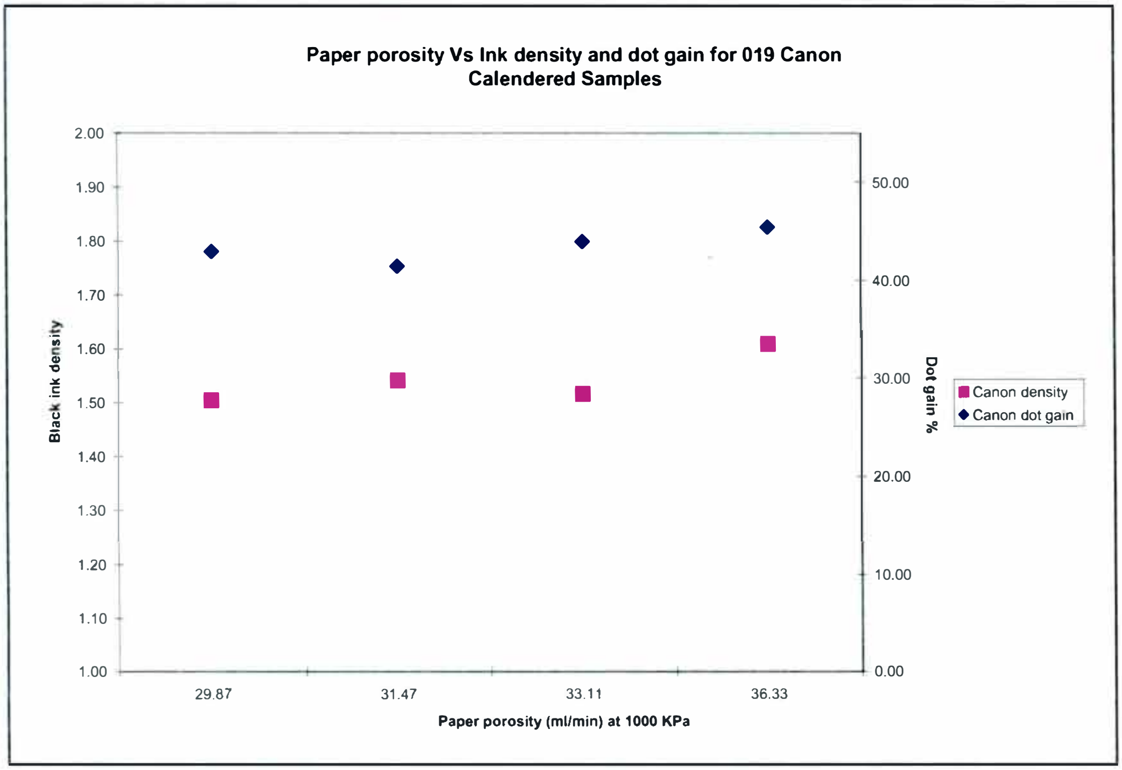


Figure 52. Paper Porosity Vs Ink Density and Dot Gain (019 Canon Calendered).

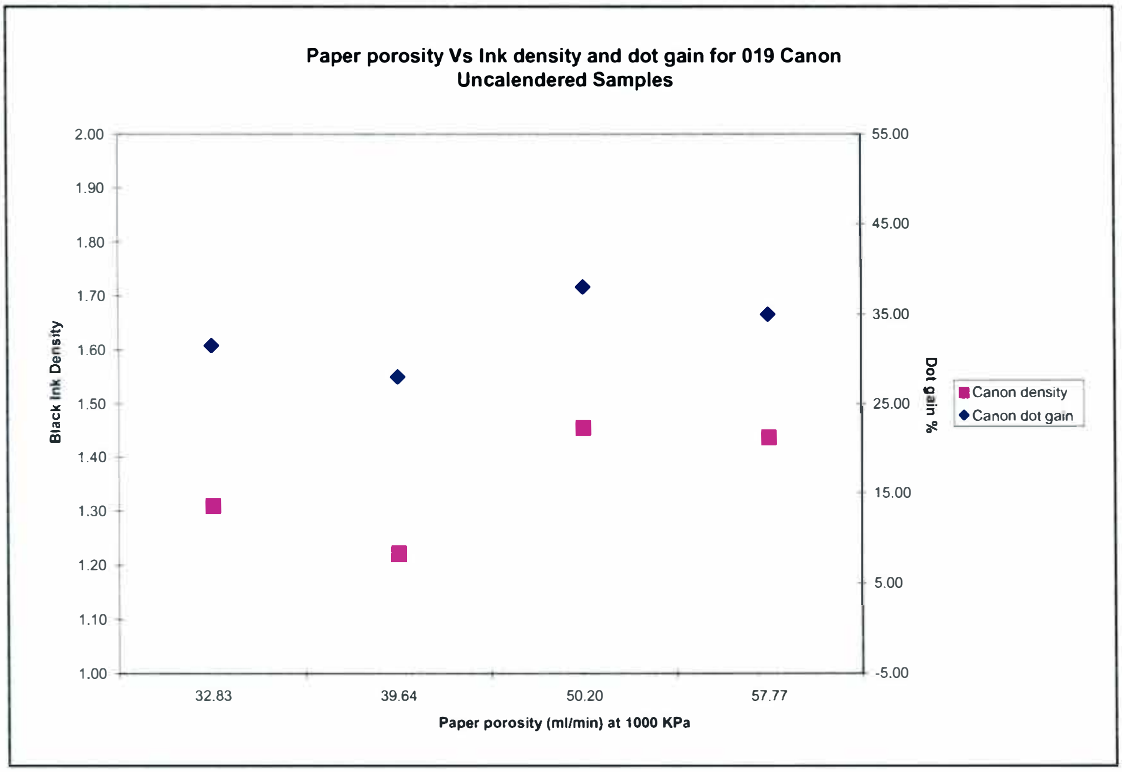


Figure 53. Paper Porosity Vs Ink Density and Dot Gain (019 Canon Uncalendered).

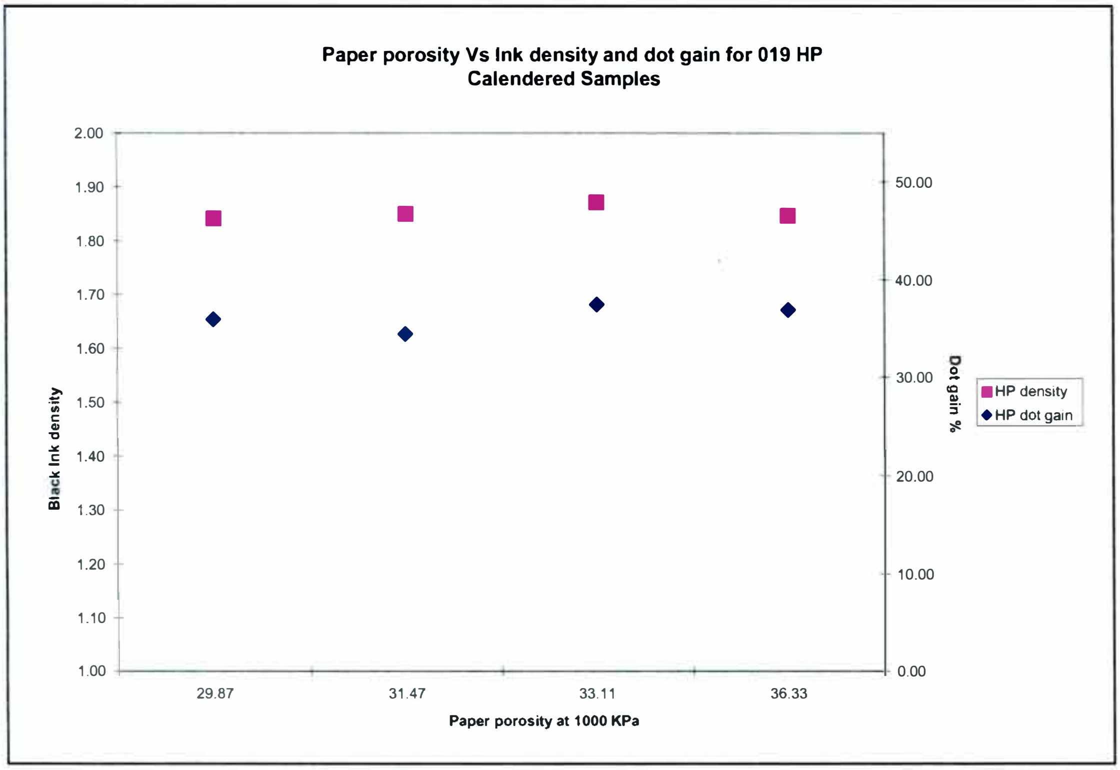


Figure 54. Paper Porosity Vs Ink Density and Dot Gain (019 HP Calendered).

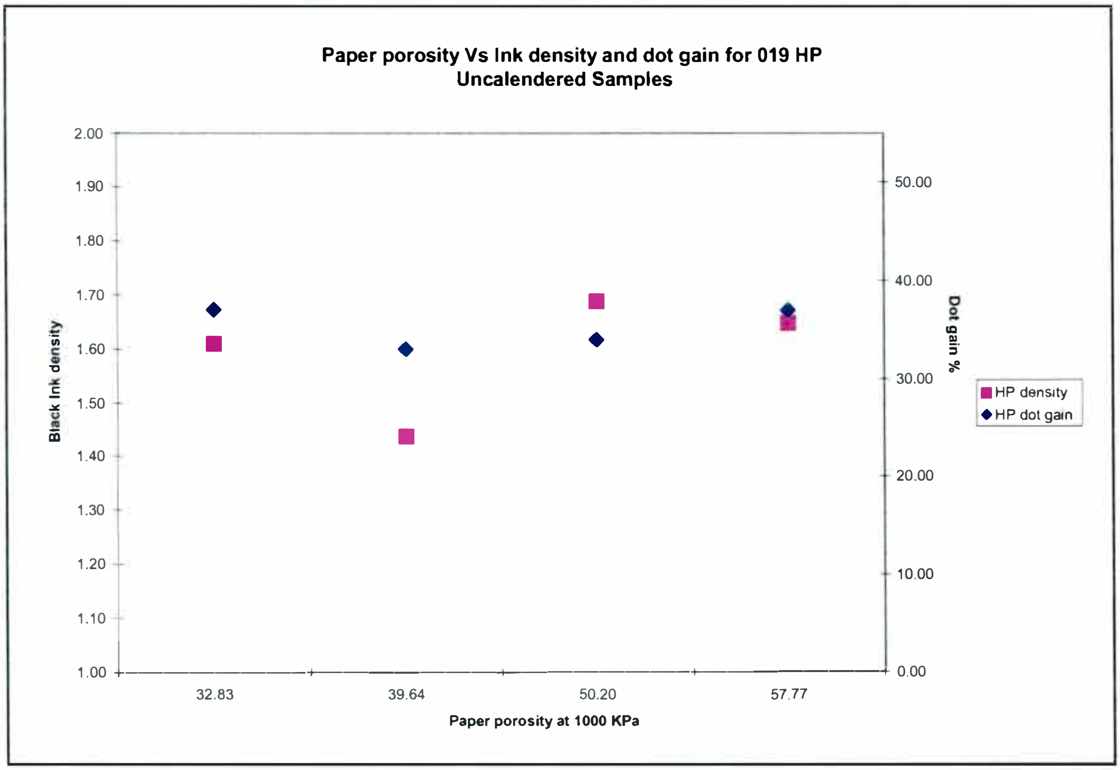


Figure 55. Paper Porosity Vs Ink Density and Dot Gain (019 HP Uncalendered).

Appendix C

Influence of Calendering on Porosity for All Coatings at 26% Solids

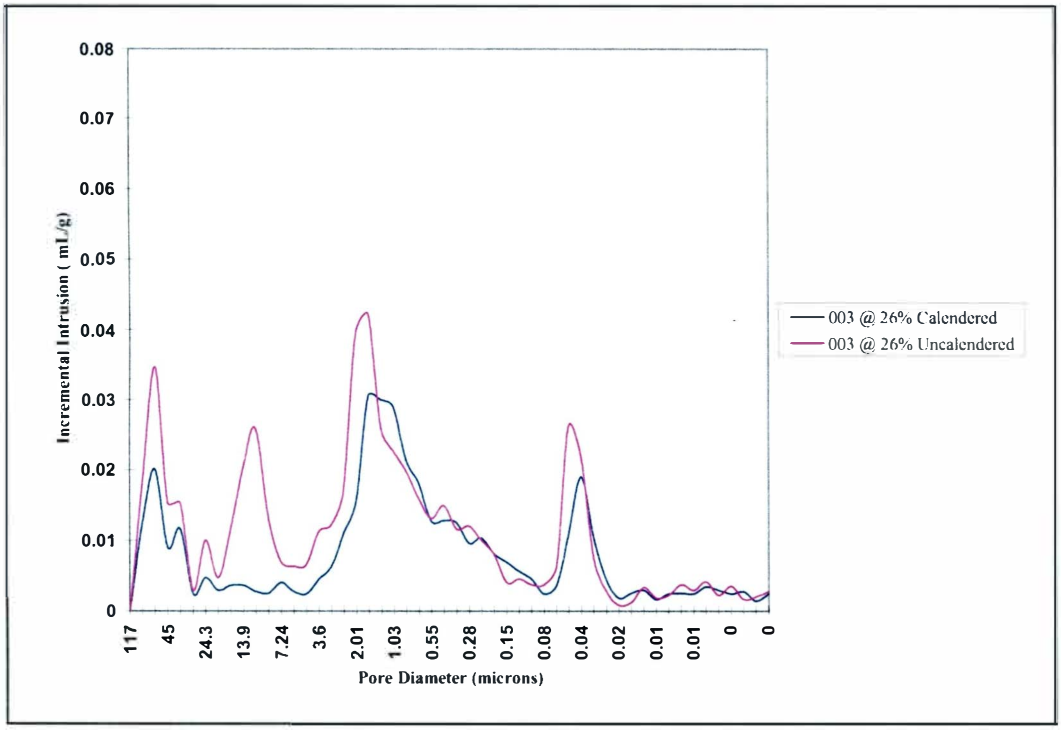


Figure 56. Influence of Calendering on Porosity of 003 Coatings Prepared at 26% Solids.

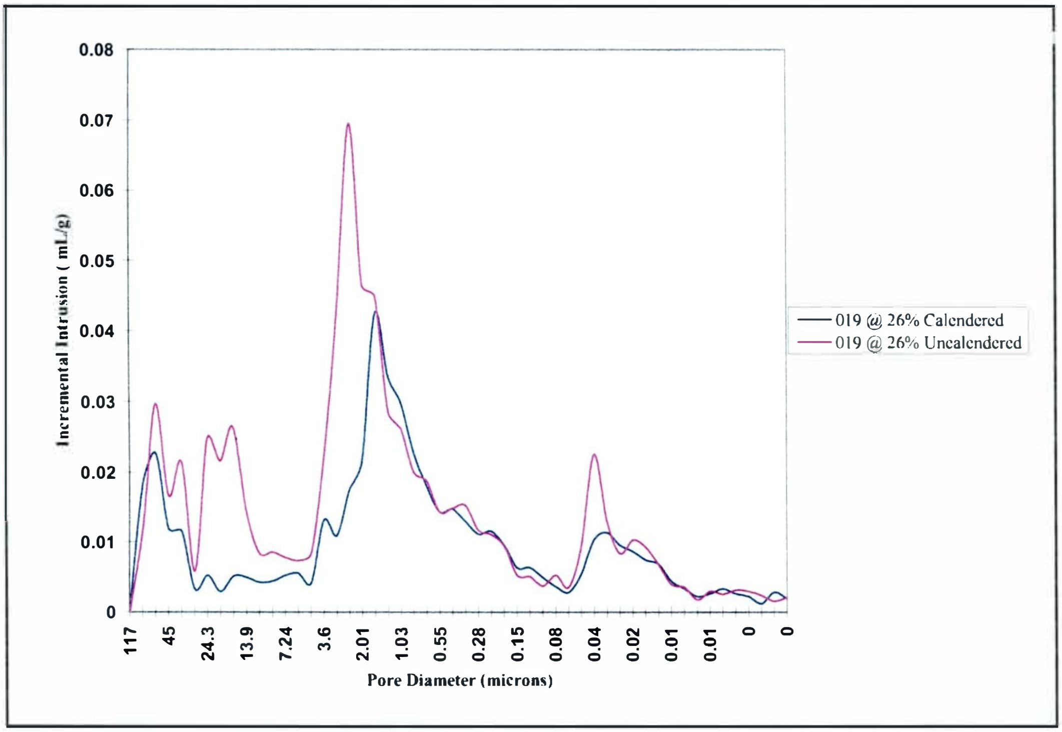


Figure 57. Influence of Calendering on Porosity of 019 Coatings Prepared at 26% Solids.

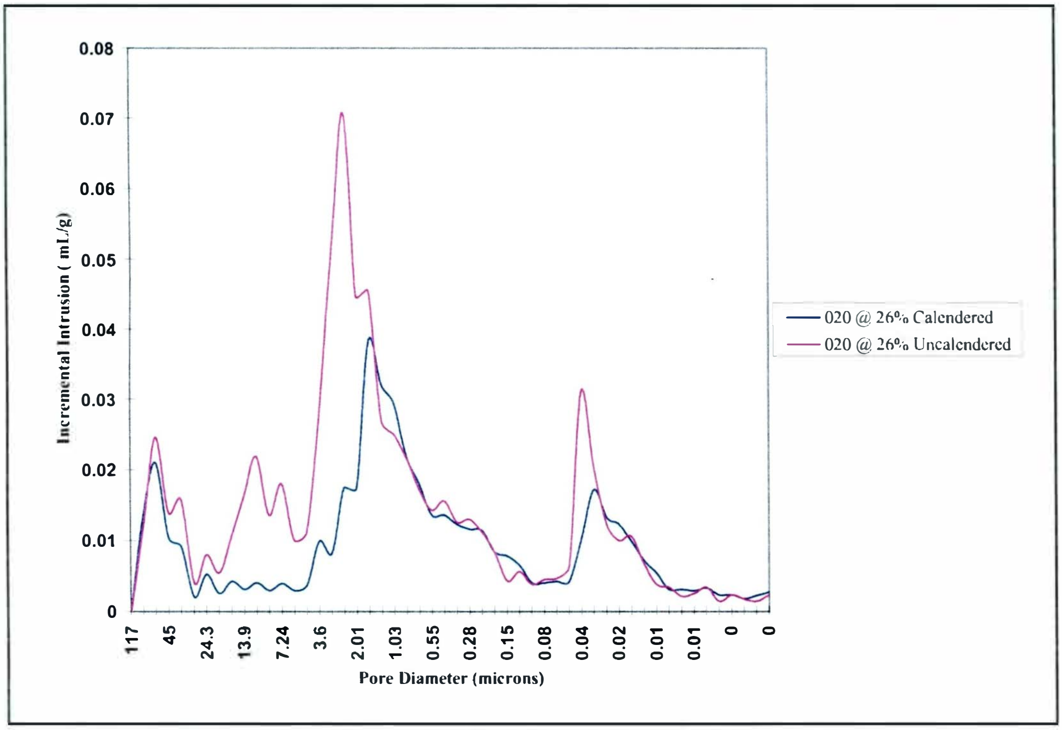


Figure 58. Influence of Calendering on Porosity of 020 Coatings Prepared at 26% Solids.

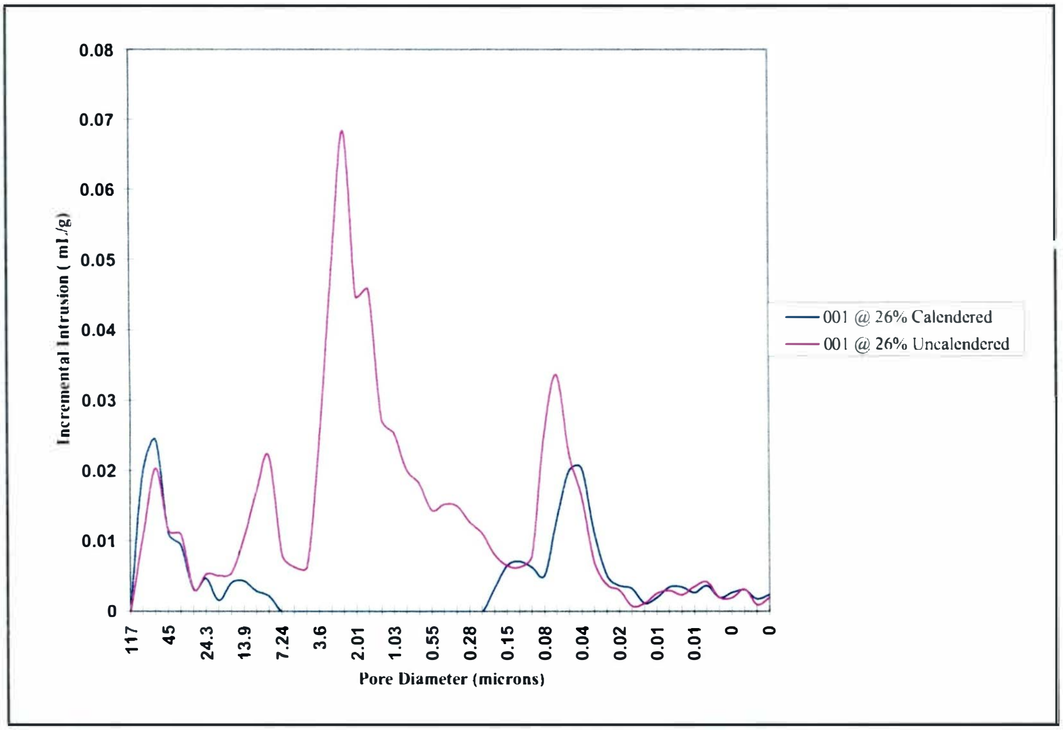


Figure 59. Influence of Calendering on Porosity of 001 Coatings Prepared at 26% Solids.

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