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**DEVELOPMENT OF A PHOTOINITIATOR BLEND FOR UV CURED INKS
AND COATINGS**

by

Madhu Bansal

A Thesis
Submitted to the
Faculty of The Graduate College
in partial fulfillment of the
requirements for the
Degree of Master of Science
Department of Paper and Printing Science and Engineering

Western Michigan University
Kalamazoo, Michigan
August 2000

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Madhu Bansal
2000

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Madhu Bansal

DEVELOPMENT OF A PHOTOINITIATOR BLEND FOR UV CURED INKS AND COATINGS

Madhu Bansal, M.S.
Western Michigan University, 2000

The purpose of this study is to develop a new standard blend of photoinitiators, having properties of low odor, high solubility, faster curing, and being efficient for pigmented as well as for clear systems. In the process, we expect to understand the behavior of mixtures of photoinitiators and their resulting UV spectra. An important first step is to prepare different blend with different photoinitiator and characterize them on cure basis. Curing properties were studied by the KMnO_4 yellow stain density test. The absorption range in the UV spectra was measured for each Photoinitiator with methanol and acrylate as background material. It was found that the acrylate esters in CN120A75/TRPGDA absorb at wavelengths up to 310 nm. Therefore, it is necessary to consider the $n \rightarrow \pi^*$ extinction coefficient, and its associated λ_{max} , and use acrylated materials as the background for optimizing the effectiveness of photoinitiators in acrylate systems. The results indicated that a combination of IR369, IR2959, IR819 and IR184 could be a solution to the problem. To find the exact proportion of each Photoinitiator in the blend, different blends were mixed with ink and samples were printed on Comco press. Ink cure analysis, Image analysis were done to determine the cure rate and print quality respectively.

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CHAPTER 1

INTRODUCTION

The printing industry changes dramatically on an almost daily basis. One of the most rapidly advancing technologies is Energy Curing, a general term that refers to ink and coatings that are cured, or hardened, by exposure to radiant energy ⁽¹⁾. This energy can be in the form of ultraviolet light or the form of accelerated high-energy electrons, concentrated into an electron beam ⁽¹⁾.

The ultraviolet printing process opens new ways of printing a wide range of new products. Many of the products produced by UV printing are superior in appearance, finish and overall effectiveness for the end user ⁽¹⁾. These products have developed new markets and created opportunities for printing companies seeking a marketing edge over their competitors. UV printed products, produced either by offset, screen printing or flexography give a sharp, strong density color reproduction, and can be complemented in production by high gloss, UV matte or special effect finishes. With UV, the printer is no longer restricted to high priced papers and boards to achieve quality print products. The higher grade coated and absorbent materials designed and manufactured specifically to suit conventional oil based inks are not essential to obtaining quality print results with UV inks and varnishes ⁽¹⁾.

UV inks print consistently on most papers and boards and practically sealed surface substrates such as plastics, vinyl, foils, acetates, etc. These materials leave the

press dry and without the production problems experienced with conventional printing inks, such as the necessity of using spray powder ⁽²⁾.

A particular advantage of printing with UV inks and varnishes over a range of different types of papers and materials is its consistency of reproduction. This is due to the fact that UV inks dry (cure) on the surface, unlike oil based inks that rely on absorbency and evaporation to assist print and drying. Therefore, with conventional inks, printed results vary substantially from coated to uncoated stocks. This is not the case with UV inks and varnishes. Whether the stock is coated or uncoated or even a sealed surface, UV inks and varnishes produce a consistent quality and visual result across this diverse range of materials ⁽¹⁾. Because of this consistency on different surfaces, customers can be offered a larger range of products on various materials and expect high quality results that are more uniform.

Reasons for Converting to UV/EB Technology

There are at least three reasons, why the producers of everyday goods have been switching to this technology recently ⁽¹⁾.

Improved Productivity

Since most systems are solvent-free and require less than a second of exposure for full cure, the productivity gains can be tremendous compared to conventional coating techniques. Printing web line speeds of 1,000 ft/min. are common and the product is immediately ready for testing and shipment ⁽¹⁾.

Suited for Sensitive Substrates

Although the pioneering work ⁽¹⁾ on these systems was primarily brought about by the need of predictable bonding between composite structures, benefits are found in bonding and coating less exotic materials. Most rad-cure systems do not contain any water or solvent ⁽¹⁾. In addition, the process provides total control of the cure temperature making it ideal for application on heat sensitive substrates.

Environmentally and User Friendly

Compositions are typically solvent-free so emissions and flammability are not a concern. Light cure systems are compatible with almost all application techniques and require a minimum of space. UV lamps can usually be installed on existing production lines. Electron beam cure setups are still primarily the province of high-tech metallurgy, however. ⁽¹⁾

Some Examples of UV/EB Cured Products

Below are lists of applications where radiation cure materials are currently used⁽²⁾. Commercial applications of radiation cure are diverse and the following is just a partial listing.

Coatings on:

1. Compact Discs
2. Hardwood Flooring
3. Shopping Bags

4. Optical Fibers
5. Furniture Laminates
6. Vacuum Metallized Plastic
7. Credit Cards
8. Beverage Cans
9. Photographic Film
10. Magazine Covers
11. Wall Paneling
12. Vinyl Floor Tile
13. Leather Finishes
14. Magnetic Media (tape & floppy disks)
15. Galvanized Metal Tubing

Disadvantages:

1. Inks and coating are more costly
2. Highly pigmented and opaque systems may be difficult to cure with UV.
3. Possible toxicology issues.
4. Limited shelf life.

Ultraviolet Curing is used to improve the quality of finished products with substantial savings in energy, time and labor⁽¹⁾. In the UV curing process, ultraviolet light interacts with specially formulated chemistries to cure coating faster and more economically than the older methods⁽²⁾. The main industrial segments for UV curing application include automotive, electrical, electronic, graphic arts⁽²⁾, rigid and flexible

packaging ^(2,4), wood ⁽⁴⁾, furniture and construction ⁽²⁾. One of the principal users of this innovative technology has been the graphic arts or printing industry ⁽²⁾. The advantages offered by this process were recognized at an early stage in development and are being utilized at an ever-increasing rate. Technical advances in inks and coatings for various print processes such as screen, flat and rotary, semi-rotary and rotary letterpress, sheetfed and web offset have brought about developments in machine design, producing presses which were not thought possible earlier. In-line processing had arrived. Curing ink by the use of UV energy is today recognized as an efficient method of obtaining dry prints directly from the press, using no spray powder.

The UV curing process requires an UV lamp ⁽⁶⁾, which directs UV light onto the formulated product ⁽¹⁾. The bulk of the formulation is made up of monomers and oligomers. Monomers are low molecular weight materials. These molecules become part of the polymer matrix in the cured coating because of their reactive functional groups. Monomers also function as diluents, used to adjust system viscosity ⁽²⁾.

Oligomers are higher weight viscous materials that determine the final properties of the coating, such as flexibility, toughness etc. These can be polyester, polyurethane, and epoxy or polyether ⁽²⁾.

The other most important component is the photoinitiator ^(4,5,7). A photoinitiator can be categorized in different ways.

According to mechanism:

1. Free radical
2. Cationic

According to its form:

1. Liquid
2. Solid

According to its absorption:

1. UV light
2. Visible light

According to its application:

1. Clear
2. Pigmented

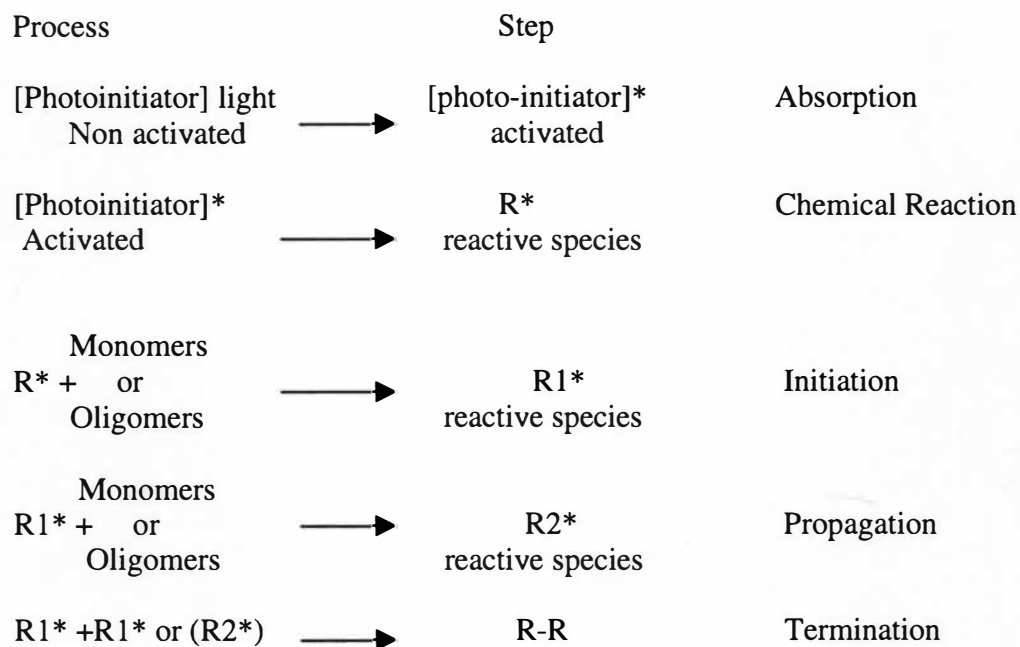


Figure1: The Role of Photoinitiators

Free radical initiators, which are the most typically used with acrylate functional resins can be described as either “hydrogen abstraction type” or “alpha cleavage type.”⁽²⁾

Hydrogen abstraction initiators have their specific uses, especially in three or four way photoinitiator blends. The alpha cleavage initiators have a generally higher efficiency, due to their generation of free radicals via a uni-molecular process, while hydrogen abstraction initiator require a hydrogen donating source in order to generate the free radicals⁽²⁾.

The other important part of this process is the UV light source⁽⁶⁾. Basically, two types of light sources are available: arc light and laser light. The arc light includes the medium pressure mercury lamp and the high-pressure xenon lamp.

CHAPTER 2

STATEMENT OF PROBLEM AND OBJECTIVES

The process of ultraviolet or UV curing is defined as hardening of a liquid film of material when exposed to ultraviolet light ⁽¹⁾. The particular substance to be processed may vary widely depending upon its application and final use, but is composed of base polymers, non-solvent diluents and photoinitiators.

The role of photoinitiator is very important in ultraviolet curing ⁽⁷⁾. It is the additive that initiates the polymerization process to quickly reach the final cross-linked product. As UV light energy is emitted, it is absorbed by the photoinitiator in the liquid, causing it to fragment into the reactive species. These species can be free radicals or cationic. The majority of systems are based on free radicals, which react with the unsaturated compounds in the formulation, and cause them to polymerize.

The main purpose of this project is to solve the problems associated with the Flint Ink's existing blend of photoinitiators, WGW14415. This blend has many problems such as:

1. It swells rollers and plates.
2. It is not highly efficient.
3. It has an odor.

I intend to test other possible combinations of photoinitiators, which may eliminate these problems. The properties that need to be examined are:

1. Solubility-This will be studied by melting the photoinitiators together and studying how the phase behavior changes with temperature and time.
2. Efficiency of Initiation-It depends on how fast the initiation occurs if two compounds are mixed together relative to the compounds alone. The yellow stain density gives some measure of the degree of curing.
3. Synergistic Effect-It depends on how two Photoinitiators react when they are put together. The main point to consider is whether they are going to activate the reaction or retard it.
4. Extinction Coefficient-By determining the extinction coefficient, we can find out the best combination for pigmented systems as well as clear systems.
5. Odor-The odor depends on the decomposition of photoinitiator during curing. The odor should be as low as possible and not objectionable.

CHAPTER 3

LITERATURE REVIEW

UV/EB curing describes the use of electron beam (EB), ultraviolet (UV) or visible light to polymerize a combination of monomers and oligomers on a substrate⁽¹⁾. The UV/EB material may be formulated into ink, a coating, an adhesive or other product. The process is also known as radiation curing or rad-cure because UV and EB are radiant energy sources⁽²⁾. The energy sources for UV or visible light cure are typically medium pressure mercury lamps, pulsed xenon lamps or lasers⁽⁶⁾. The coatings cured with these light sources are usually clear or translucent, although thin opaque coatings are also possible. Electron beam accelerators are used to generate the electron stream capable of curing thicker, pigmented coatings. Unlike photons of light, which tend to be absorbed mainly at the surface of materials, electrons have the ability to penetrate through matter⁽¹⁾.

Early History

The concept of using ultraviolet light to achieve rapid or instant drying is one, which has been developed over many years. Although there have been earlier claims for its beginning in research and development work, a patent to convert a liquid to a solid film was not granted until the 1940's.

Early Applications

Early use of the new technology was in the area of wood printing using extended printing lines having multiple banks of low powered lamps. By today's standards, these lines were not very efficient because of the slow speeds then possible. They did show, however, that problems such as space requirements and solvent pollution could be solved using the new process. The development and subsequent introduction of the medium pressure mercury vapor lamp gave rise to interest from the printing trade in the area of heat-set web offset inks because of greater curing speeds being possible. Pressure to do something about the quantity of pollutants being released into the air was also being experienced by this section of the trade. This step forward caused others in allied areas to reflect on what the advantages for other facets of printing would be. Trials for various types of printing were initiated and resulted in machines being redesigned or adapted to take advantage of UV curing⁽¹⁾.

Over the years a great deal of development was done, to improve the characteristics of the UV cured products. Much time and effort were devoted to improving the inks to make them perform more like conventional ones. Early UV inks had a great tendency to exhibit dot gain, some colors being worse than others. Technology has advanced to the point where it is now extremely difficult to determine the difference between the two inks. Printing characteristics have also improved considerably⁽¹⁾.

Components of UV/EB Formulations

Oligomers

The overall properties of any coating, ink, adhesive or binder cross-linked by radiant energy are determined primarily by the oligomers used in the formulation. Oligomers are moderately low molecular weight polymers, most of which are based on the acrylation of different structures. The acrylation imparts the unsaturation or the "-C=C-" group to the ends of the oligomer^(4,5).

Monomers

Monomers are primarily used as diluents to lower the viscosity of the uncured material to facilitate application. They can be mono-functional, containing only one reactive group or unsaturation site, or multifunctional. This unsaturation allows them to react and become incorporated into the cured or finished material, rather than volatilizing into the atmosphere as is common with conventional coatings. Multifunctional monomers, because they contain two or more reactive sites, form links between oligomer molecules and other monomers in the formulation^(4,5).

Photoinitiators

These ingredients absorb light and are responsible for the production of free radicals or cations. Free radicals or cations are high-energy ions that induce crosslinking between the unsaturation sites of monomers, oligomers and polymers.

Photoinitiators are not needed for electron beam cured systems because the electrons themselves are able to initiate cross-linking^(4,5).

Additives

The most common are stabilizers, which prevent gelation in storage and premature curing due to low levels of light exposure. Color pigments, dyes, defoamers, adhesion, promoters, flattening agents, wetting agents and slip aids are examples of other additives, depending on application^(4,5).

Curing Equipment

The equipment used to produce a cure in the previously mentioned applications may fall into one of several categories depending upon available space, cure speed and other considerations. Successful light curing requires matching an adhesive to the light source. Frequently, assemblers inherit a light-curing system and assume it's the correct one for their specific application—until they run into problems achieving cure. Light-curing equipment must be carefully selected to provide optimal curing parameters. An adhesive that requires a high-intensity light source cannot be cured with a low-intensity lamp⁽⁶⁾.

Lamps in Industrial Use

Black Light Lamps

They are fluorescent lamp designed to emit UV-A wavelengths. Irradiance is low—about 6 to 10 milliwatts per square centimeter—limiting its use in a production environment. However, these lamps can be used for quality control purposes or with materials that cure under low-energy radiation. Spectral output maximizes at 320 to 400 nanometers⁽⁶⁾.

Electrode-Type Mercury Vapor Lamps

They are available in sizes of a few inches to 80 inches, and at power levels of 100 to 600 watts per inch. The most popular lamps are 200 to 300 watts per inch (78-118 watts per cm). The lamps produce a radiation of 100 to 200 milliwatts per square centimeter. They can't be turned on and off instantly, but can be switched to one-half or one-third power and then instantly raised to full power. The intensity of an electrode-mercury vapor lamp deteriorates with use. After 1,000 hours, the output is reduced 15 to 25 percent. Because lamp deterioration is seldom immediate or severe, it is not easily perceptible. Regular monitoring of lamp intensity at the working distance is extremely important⁽⁶⁾.

High Pressure Mercury Lamps

These lamps are less common in the United States, are often used in wand systems⁽⁶⁾. These spherical lamps are of the electrode type, but feature portability and fast start-up time.

The Medium Pressure Mercury Vapor Lamp

Medium-pressure mercury vapor lamps with electrodes—also known as mercury arc lamps—combine cure efficiency with versatility and moderate cost. Intense emissions occur in the 240 to 270 nanometers and 350 to 380 nanometers ranges, where typical UV photoinitiators absorb energy. This lamp is made in various lengths to suit the machine or application, and is a quartz tube about 1" or 25mm in diameter. An inert gas, such as argon or xenon, together with a small amount of mercury is used to fill the tube, which also has an electrode, installed at either end. When the lamp is connected to an appropriate power source, an electrical arc passes between the two electrodes vaporizing the mercury. The resultant energy emission from the lamp is white light, infrared and ultraviolet. Lamps are available to suit almost any application and power requirement. The medium pressure mercury lamp is currently the overwhelming industry choice because of its high power (200-700 watts/inch) and important emission lines, which are absorbed by the most commercially available photoinitiators. Regardless the type of light source, however, the emission spectra of the lamp must overlap the absorption spectrum of the chosen initiator⁽⁶⁾.

Electrode-Less Lamps

The electrode-less mercury vapor lamp is a medium-pressure lamp where the mercury in a quartz sleeve is vaporized by microwave energy rather than an electrode process. This type of lamp has a similar spectral emission to the electrode type but the method of operation is totally different. Microwaves generated by magnetrons rather than an electrical arc energizes the lamp in operation. The tube may also be filled with other materials depending on the spectral emission required for the application. Major advantages from lamps with electrodes include instant on-off capability, bulb life of 3,000 to 5,000 hours and bulbs with smaller diameter. With electrode-less mercury vapor lamps, irradiance well in excess of 150 milliwatts per square centimeter can be generated on a working surface, enabling very high cure speed. Adding metal halide to the contents of a lamp changes the spectral output of these bulbs. Disadvantages of electrode-less bulbs include significantly higher cost per bulb and bulb length sizes of only 6 or 10 inches. Placing bulbs in tandem can compensate for bulb length. Metal-halide lamps are a type of medium-pressure mercury arc lamp with electrodes. The spectral output is modified by the addition of metal halides to the bulb contents. The most common additive is an iron halide that enhances output in the UV region. These lamps are most often used in low- to medium-energy equipment. Pulsed-xenon and flash-xenon lamps use xenon gas and no mercury to generate UV, visible and infrared radiation. This equipment generates extremely high energy and is often used to cure fiber-optic coatings. Varieties of lamp configurations are possible, although high cost—higher than electrode-less systems—is a disadvantage^(6,8).

CON-TROL-CURE® Ink-Cure Analyzer™

The Ink-Cure Analyzer™ is a computer-based instrument that measures the exact amount of cure on the finished product, giving us a view into the workings of the photo-polymer process⁽⁸⁾

Imagine having a charted profile of the near cure, overcure or undercure condition of UV inks, coatings, adhesives and other crosslinkable materials. That's exactly what the Ink-Cure Analyzer™ produces. What we have here is a device that can identify a good product from a bad product. It gives vital information on what has happened during the curing of that product, a culmination of the process. Therefore, in addition to controlling all the production variables, you now have the ability to scientifically evaluate the results.

On a multi-color job, operators can measure the parameters and assure themselves that they are doing the job properly at each step, rather than discover at the end that something went wrong. The faster a job is printed, the more waste is made if things aren't right... the need for controls is very strong."

The success rate with the Ink-Cure Analyzer™ has been 100%.⁽⁸⁾

Inks

Although great advances have been made in the conventional ink sphere, a process depends largely on air-drying. UV cured inks, on the other hand, remain open in the duct and on rollers. Curing only occurs on the substrate when ink is exposed to UV energy generated by the machine's UV lamp array^(1,7).

Process Suitability

Curing ink by UV allows the printer to expand into areas that previously would have presented many difficulties. One instance is in the production of plastic products. As the inks are cured virtually instantaneously, no spray powder is required. This results in the product feeling smooth, not gritty to the touch. Lack of spray powder also has a bearing on another benefit of UV cure, that of high scuff-resistance. This highly sought-after feature is synonymous with the production of an overwhelming number of cartons for many quality products. Products that require high scuff resistance, such as wine and other labels, are almost exclusively printed with UV cured coatings and inks. The range of products to suit various applications is quite comprehensive and can be tailored to suit stringent requirements ⁽¹⁾.

Space Saving

As the products are cured almost immediately, the prospect of saving space is great. Space can be utilized more economically than normal due to higher machine piles, faster turn-around for partly finished products and speedier conversion of products. Short deadlines become more easily achievable. Many companies could benefit from not having to wait for inks to set and dry on the short runs which are now part of the overall printing scene. Scratching is less of a problem on back-up work, as is the build-up, which is often so prevalent on short-run, backed-up work.

Viewed as an essential component of today's printing scene, UV curing can be

used to eliminate "dead-time" in the press room and also other areas, like converting and final packing⁽¹⁾.

Environmentally Sound

Conservation of the atmosphere is increasingly becoming an issue for most businesses. Many processes used at present release massive quantities of solvents into the air. Evidence shows that the use of volatile solvents can have long term adverse health effects. UV cured materials do not release solvents into the air. Strict guidelines apply to the manufacture of UV products to ensure safe usage by print personnel. Materials used have an extremely low impact on the human body. Accidental ingestion of UV ink has been shown to have a non-cumulative effect. Further reference to this above aspect can be found under the heading "Health and Safety"⁽¹⁾.

Quality Control

Problems that occur while using conventional inks due to drying are eliminated when UV curing is employed. Results of this are shown in maintaining closer control over the color on the job in hand. Color variation due to individual interpretation of color dry-back is removed⁽¹⁾.

Stock Suitability

Using normal parameters and conventional printing methods, it has become an accepted norm that a top quality production requires the use of the best material

available. This is largely due to the absorption of part of the ink into the substrate.

Technical advances in the paper-manufacturing field have produced materials with excellent working characteristics. Color rendition is usually brilliant on these materials. Substrates in this category are usually priced accordingly at the upper end of the price range, as a top quality product normally would be. Instead of being in the situation of most users of quality stock, the UV printer is able to utilize a good stock which can be considerably less expensive. This does not mean that the final product is less attractive; in fact, the opposite is usually the case. Almost all of the credit for this situation is because in UV printing the print remains on the surface of the substrate, and does not become absorbed by it. Comparison of UV and conventional prints on the same lower grade stock show a pronounced increase in quality and color on the UV print. Further, coating the product with a UV cured coating visually enhances the print while also substantially increasing the body of the substrate ⁽¹⁾.

Energy Savings

Curing materials using UV is an efficient method that can lower production costs. Apart from the "dead-time" elimination during production there is also the lack of press cleaning which involves cleaning the spray powder from gripper bars and other internal and external parts. This is a normal and necessary part of press maintenance with conventional printing. Electrical power usage is also lower with normal UV curing than a typical IR lamp, hot/air-drying system ⁽¹⁾.

Photoinitiators

IR369

Irgacure 369, manufactured by Ciba Chemicals, is an excellent alpha amino acetophenone photoinitiator for non-white pigmented systems. It has strong absorption where several important emission wavelengths from mercury vapor lamps are available⁽⁷⁾.

Chemical Structure: 2-benzyl-2-N,N-dimethylamino-1-(4-morpholinophenyl)-1-butanone(DBMP)

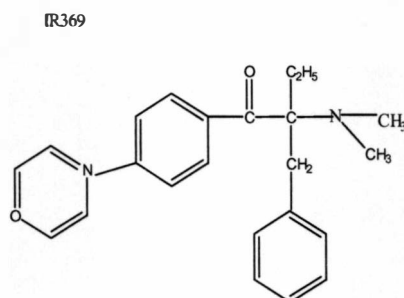


Figure 2: Chemical Structure of IR 369

Table 1

Physical Properties of IR 369

Appearance	Slightly Yellow Powder
Molecular Weight, g/mole	366.6
Melting Range, °C	110-114
Density, g/ml	1.18

Table 2
Extinction Coefficients of IR 369

Wavelength (n m)	Coefficient (ml gm ⁻¹ cm ⁻¹)
254	7.470×10^3
302	3.587×10^4
313	4.854×10^4
365	7.858×10^3

IR369 is one of the photoinitiators that exhibits low volatility. IR369 tends to show better through cure than IR907 in dark color pigmented systems. It has its absorption maximum red shifted to make more efficient use of 365nm-emission line from Hg vapor lamp. IR369 is good for pigmented systems and can be used as a secondary component for visible systems. Addition of IR184 or IR1173 is recommended along with IR369 to balance surface cure and body cure ⁽⁷⁾.

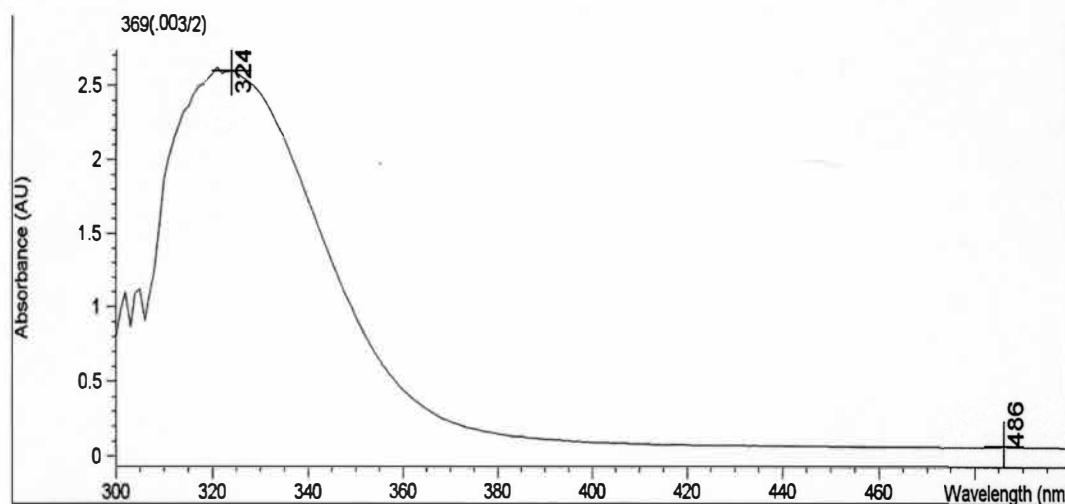


Figure 3: Absorption Spectrum of IR 369

IR1173

It is a good liquid photoinitiator, manufactured by Ciba, for use in a wide range of UV curing formulations. It has good solvency properties and can act as a solvent for other photoinitiators.

It can act as a solvent for other photoinitiators. It can also act as a diluent for viscosity reduction.

Chemical Structure: 2-hydroxy-2-methyl-1-phenyl-propan-1-one

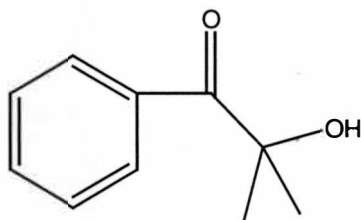
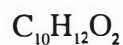


Figure 4: Chemical Structure of IR 1173

Table 3

Physical Properties of IR1173

Appearance	Clear, Colorless to light yellow liquid
Molecular Weight, g/mole	164.2
Melting Range, °C	80-81 @ 1mm Hg, 250°C
Density, g/ml	1.074-1.078
UV absorption Peaks, nm	265-280, 320-335

Table 4
Extinction Coefficients of IR1173

Wavelength (n m)	Coefficient (ml gm ⁻¹ cm ⁻¹)
254	4.064×10^4
302	8.219×10^2
313	5.639×10^2
365	7.388×10^1

IR1173 can reduce the viscosity of a clear coating by about 35.5%, while IR184 gives 27.5% reduction in viscosity⁽⁷⁾.

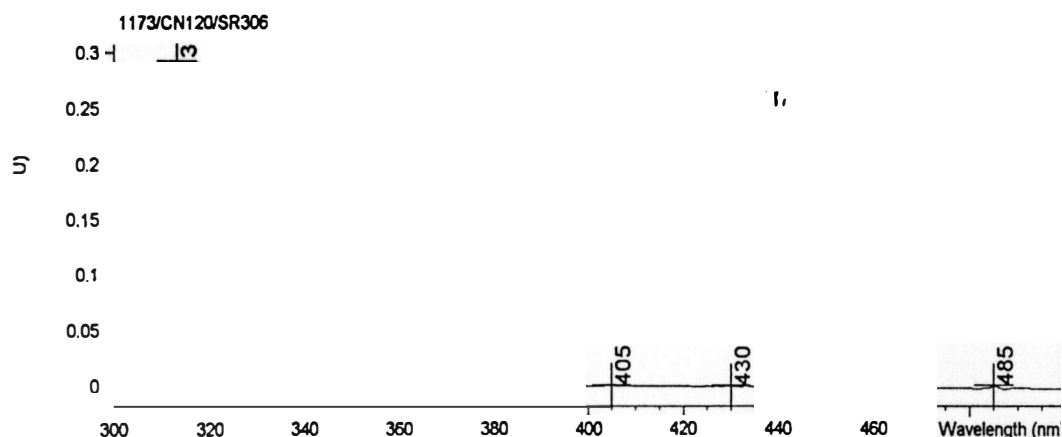


Figure 5: Absorption Spectrum of IR 1173

IR 2959

Irgacure 2959, manufactured by Ciba Chemicals, is a hydroxy ethoxy derivative of darocure 1173. The hydroxy functional group enhances the

compatibility of IR2959 in UV curable water borne systems. It offers the lowest odor in cured films, compared to other photoinitiators.

4-(2-hydroxyethoxy) phenyl- (2-hydroxy-2-methylpropyl) ketone. $C_{12}H_{16}O_4$

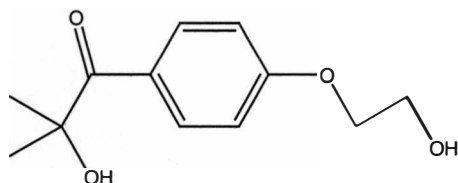


Figure 6: Chemical Structure of IR 2959

Table 5

Physical Properties of IR 2959

Appearance	white crystals
Molecular Weight, g/mole	224.26
Melting Range, °C	86.5-89.5
UV absorption Peaks, nm	320-330, 275-285

Table 6

Extinction Coefficients of IR 2959

Wavelength (n m)	Coefficient (ml gm ⁻¹ cm ⁻¹)
254	3.033×10^4
302	1.087×10^4
313	2.586×10^3

Table 6 – Continued

365

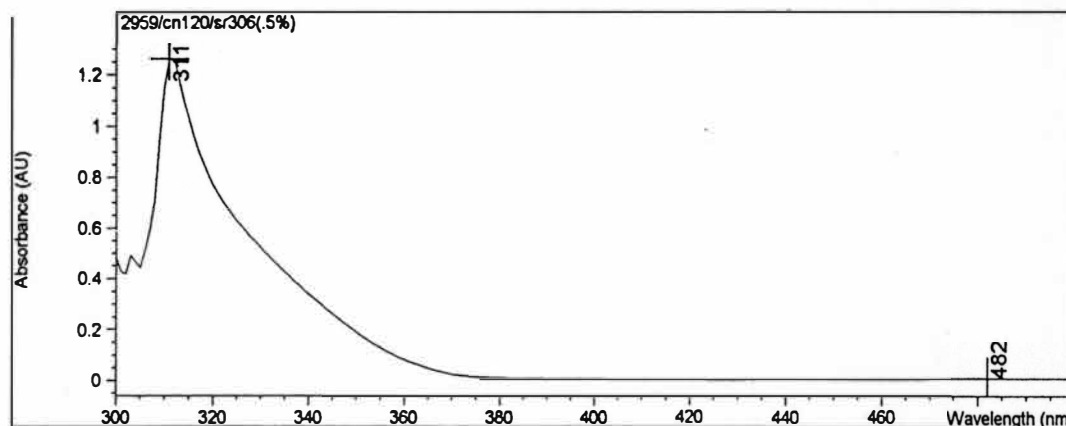
 4.893×10^1 

Figure 7: Absorption Spectrum of IR 2959

IR 2959 can be used for clear systems ⁽⁷⁾.

IR1700

This is a liquid photoinitiator, manufactured by Ciba Chemicals, useful in curing pigmented coatings and inks. Its photo bleaching effect makes the photoinitiator suitable for white coatings and inks. It has good absorption between 350 nm, therefore it provides good through cure in highly pigmented white systems ⁽¹⁾.

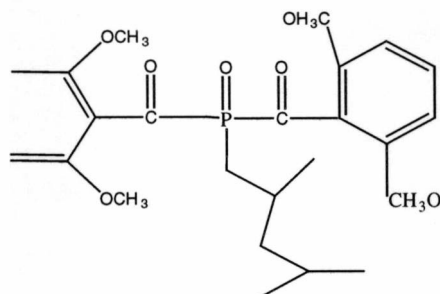


Figure 8: Chemical Structure of IR 1700

This is a mixture of DMBAPO and HMPP.

DMBPO: 25% bis(2,6-dimethoxybenzoyl)-2-4-4-trimethylpentyl phosphine oxide. $C_{26}H_{35}O_7P$

HMPP: 75% 2-hydroxy-2-methyl-1-phenyl-propan-1-one. $C_{10}H_{12}O_2$

Table 7

Physical Properties of IR 1700

Appearance	clear, light yellow liquid
Molecular Weight, g/mole	196.94
Melting Range, °C	liquid at room temperature
UV absorption Peaks, nm	245, 325

Table 8

Extinction Coefficients of IR 1700

Wavelength (n m)	Coefficient (ml gm ⁻¹ cm ⁻¹)
254	3.207×10^4
302	5.750×10^3
313	4.162×10^3
365	8.316×10^2

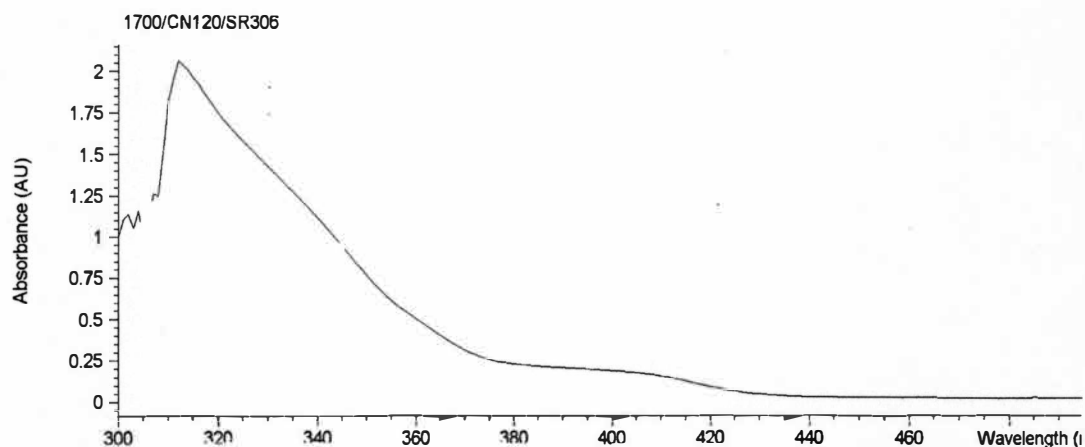


Figure 9: Absorption Spectrum of IR 1700

IR184

Irgacure 184 is a highly efficient photoinitiator, manufactured by Ciba Chemicals, developed for UV curing coatings, inks and adhesives. It is recommended when non- yellowing properties are critical. It also has a relatively low odor⁽⁷⁾.

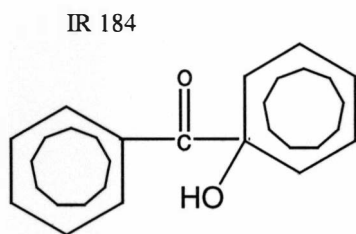


Figure 10: Chemical Structure of IR 184

Chemical Structure: 1-hydroxycyclohexyl phenyl ketone. $C_{13}H_{16}O_2$

Table 9

Physical Properties of IR 184

Appearance	White granular powder
Molecular Weight, g/mole	204.27
Melting Range, °C	44 – 49
UV absorption Peaks, nm	240-250, 325-330

Table 10

Extinction Coefficients of IR 184

Wavelength (nm)	Coefficient (ml gm ⁻¹ cm ⁻¹)
254	3.317×10^4
302	5.801×10^2
313	4.349×10^2
365	8.864×10^1

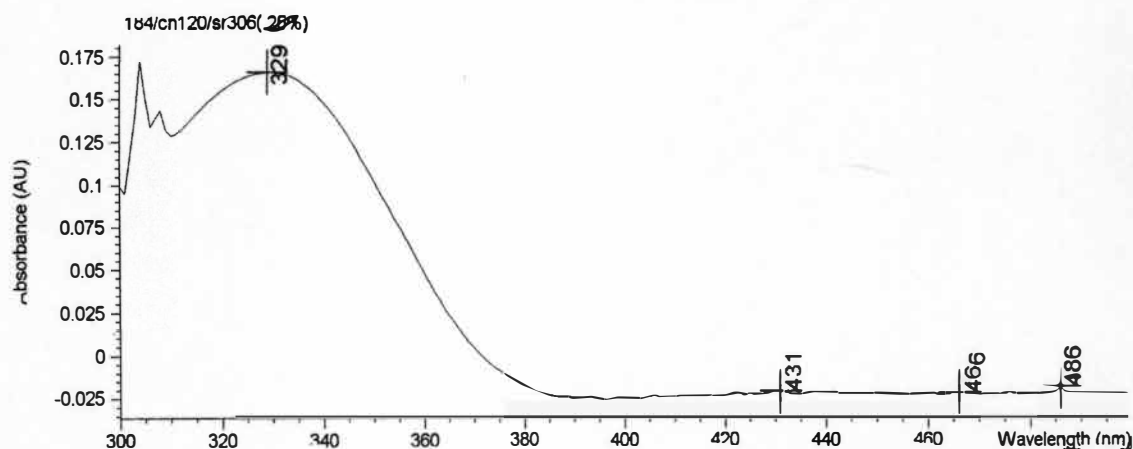


Figure 11: Absorption Spectrum of IR 184

MBB

Methyl o-benzoyl benzoate also known as Photocure-55, manufactured by Ciba Chemicals. It acts as a photosensitizer for UV-curable inks, coatings. Adhesives, resins, UV absorber, soluble in the usual organic solvents. It is insoluble in water⁽⁷⁾.

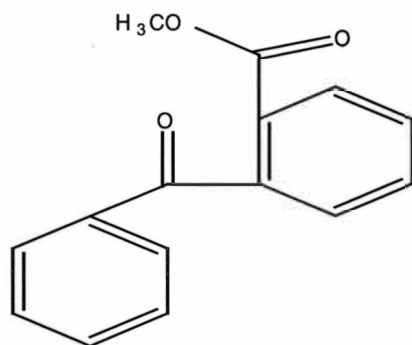


Figure 12: Absorption Spectrum of MBB

Chemical Structure: Methyl o-benzoyl benzoate. $C_{15}H_{12}O_3$

Table 11

Physical Properties of MBB

Appearance	White to off-white crystalline powder
Molecular Weight, g/mole	240.26
Melting Range, °C	48-54
UV absorption Peaks, nm	240-250, 325-330

ITX

ITX is a highly efficient UV curing agent, manufactured by Ciba Chemicals, for use as an initiator in the photo-polymerization of photo reactive polymer systems⁽⁶⁾.

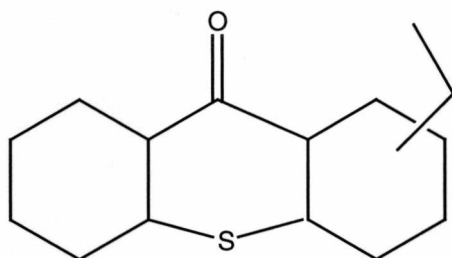


Figure13: Chemical Structure of ITX

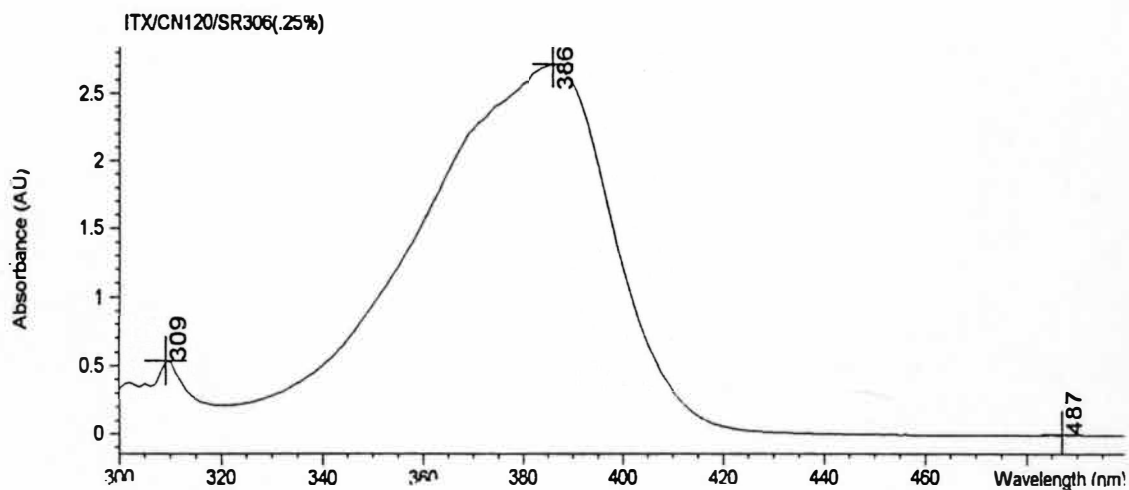


Figure14: Absorption Spectrum of ITX

Table 12
Physical Properties of ITX

Appearance	Yellow powder
Density, g/ml	1.074-1.078
Melting Range, °C	57- 72
UV absorption Peaks, nm	260-265

BISDEABP

Ethyl Michler's ketone, also known as 4,4-bis (diethylamino)benzophenone , manufactured by Ciba Chemicals ⁽⁶⁾.

Table 13
Physical Properties of BISDEABP

Appearance	Gray to Light Yellow Powder
Density, g/ml	1.074-1.078
Melting Range, °C	93-95
UV absorption Peaks, nm	260-265

IR651

Irgacure 651, manufactured by Ciba Chemicals, is a good photoinitiator for general applications where non- yellowing is not important. It can be used for wood, metal and plastic coatings. It offers good storage stability over benzoin ethers ⁽⁷⁾.

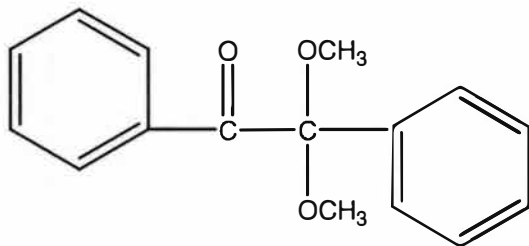


Figure 15: Chemical Structure of IR651

2,2-dimethoxy-2-phenyl acetophenone (BDK). $C_{16}H_{16}O_2$

Table 14

Physical Properties of IR 651

Appearance	White Crystalline Powder
Density, g/ml	1.212
Melting Range, °C	63-66
UV absorption Peaks, nm	330-340

Table 15

Extinction Coefficients of IR 651

Wavelength (n m)	Coefficient (ml gm ⁻¹ cm ⁻¹)
254	4.708×10^4
302	1.671×10^3
313	7.223×10^2
365	3.613×10^2

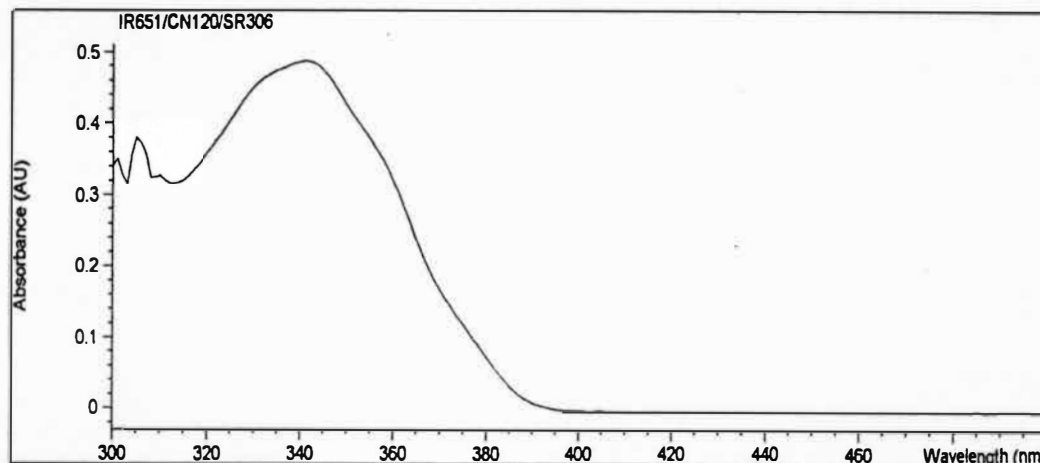


Figure 16: Absorption spectrum of IR 651

A general purpose initiator. It is useful for curing unsaturated polyester resins.

IR651 can be used as a secondary element for pigmented and white systems.

IR819

Table 16

Physical Properties of IR 819

Appearance	Light yellow powder
Molecular Weight, g/mole	417.97
Melting Range, °C	131-135
UV absorption Peaks, nm	360-365,405

IR819, manufactured by Ciba Chemicals, has wide absorption in the long wavelength, also has very strong absorption in near UV range. IR819 is mostly soluble in dichloromethane ⁽⁷⁾.

Table 17
Extinction Coefficients of IR 819

Wavelength (n m)	Coefficient (ml gm ⁻¹ cm ⁻¹)
254	1.953×10^4
302	1.823×10^4
313	1.509×10^4
365	2.309×10^3

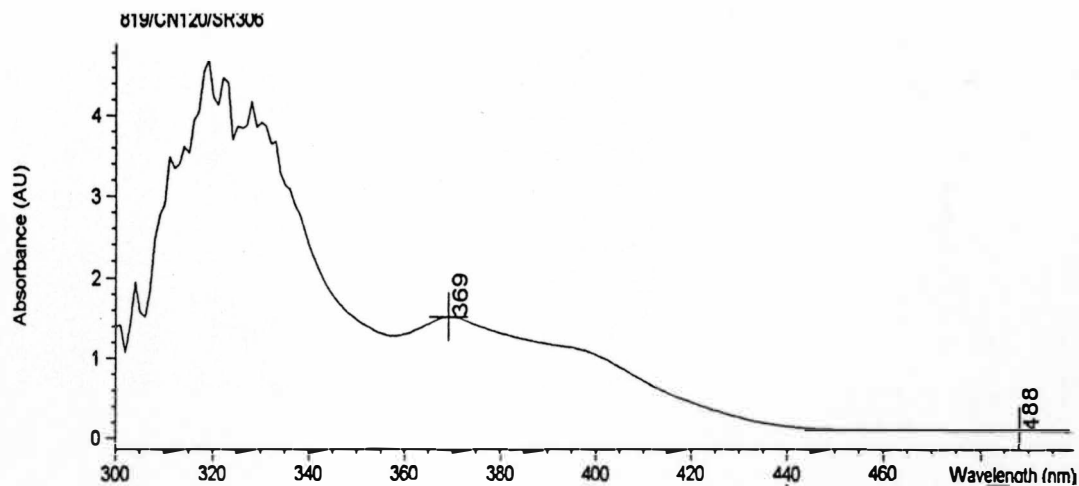


Figure 17: Absorption Spectrum of IR 819

KIP150

It is a polymeric hydroxy alkyl phenone, manufactured by Ciba Chemicals. It is a unimolecular photoinitiator. KIP150 is polymeric in nature with low odor, low migration and low extractable ⁽⁷⁾.

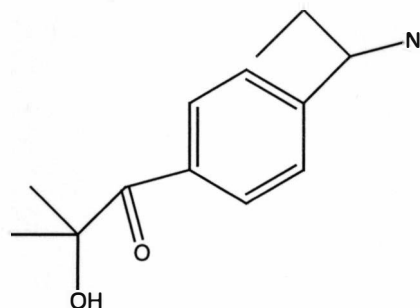


Figure18: Chemical Structure of KIP150

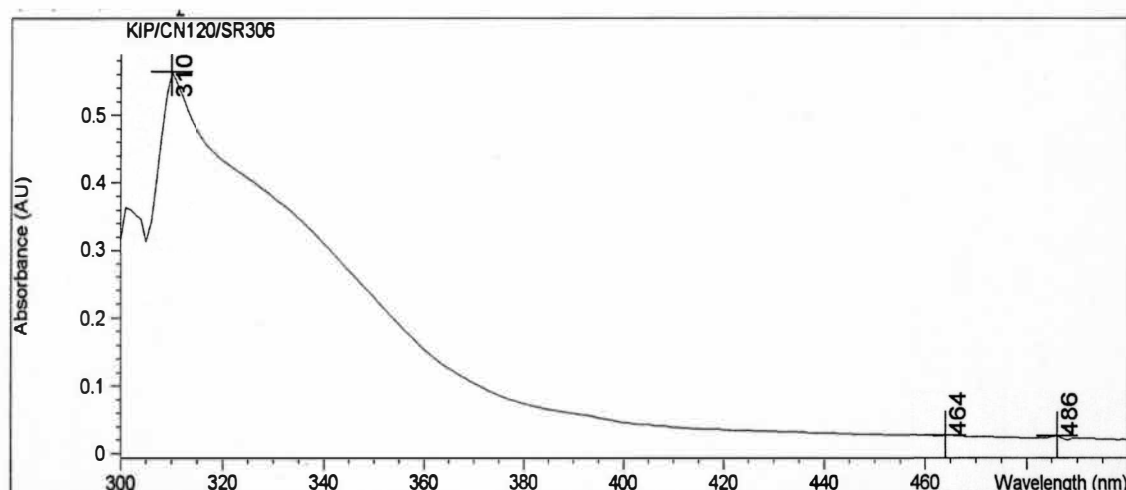


Figure 19: Absorption Spectrum of KIP150

P115

P115, manufactured by Ciba Chemicals, is a reactive amine additive used to increase the cure speed of ultraviolet light cured coatings in place of more fugitive amine synergists. It can increase the cure speed if mixed with other photoinitiators.

Recommended levels are 5 to 15% based on the total formulation, usually combined with 5% benzophenone. It has low color, low viscosity and low volatility.

P115 is recommended as a co-initiator for over prints varnishes, pigmented coatings, clear coatings on paper and plastics, silk screen and Flexo inks. Coating based on P115, cure faster in comparison to other coatings ⁽⁷⁾.

CHAPTER 4

EXPERIMENTAL DESIGN AND METHODOLOGY

Phases

The experimental study is divided into five phases:

1. Phase I: Preparation of photoinitiator blend.
2. Phase II: Photoinitiator blend characterization.
3. Phase III: Comco run & Print Evaluation.
4. Phase IV: Thesis write up.

Phase I

In this phase the photoinitiator blends were prepared at Flint inks, radiation cure lab. The photoinitiator used were IR1850, IR2959, IR369, IR184, TPOL, TPO, BISDEABP, KIP150, IR1700, IR1173 and ITX. This phase consist of preparing photo-initiator blends, making Drawdown using anilox, testing them for curing properties with different inks and substrates. The UV spectrum for each blend will be drawn by using a UV/VIS spectrophotometer. The curing analysis will be done by using curing equipment (CON-TROL-CURE® Ink-Cure Analyzer™). The blends will also be tested for odor and mutual solubility. Degree of curing will be measured by using KmnO_4 yellow stain density test. On the basis of blends of two photoinitiator

that were curing at 100 mJ, blends of three photoinitiator were made and degree of curing will be measured for further testing.

Phase II

This involves determining the optimum photoinitiator ratio in the blend. Based on the results from phase 1, blends with good curing, lower odor and higher solubility will be separated out for further experiments. All blends which are not chemically compatible (based on their chemical structures and cure analysis) will be discarded. Further testing will be done with different composition of photoinitiators (based on the overall reactivity of particular photoinitiator in the blend). Concentration of an individual photoinitiator can be decided, depending on their chemical and physical behavior using molar ratio. Final blends (may be 4 or 5 in number) will be prepared to get all the desired properties like surface cure, through cure, low odor also the low cost.

Phase III

In this phase, the blend will be mixed with Black base made of black pigment, CN12A75 and SR306. Samples will be printed using the COMCO Commander Flexographic press and then final curing analysis will be done to determine the viability of the photoinitiator blend as the most suitable blend for clear as well as pigmented systems. The quality of print will be determined by printability analysis.

Table 18

Test Equipment and Method

Name of the Test	Test Equipment
Energy of Cure	Con-Trol-Cure® Compact Radiometer # M007-008
Gloss	Hunter 75 ⁰ Glossmeter
Solid Density	X-Rite 418 Reflection Densitometer
Absorption Spectrum	UV Visual Spectrophotometer

Phase IV

In this phase the thesis writing and thesis defense will be done.

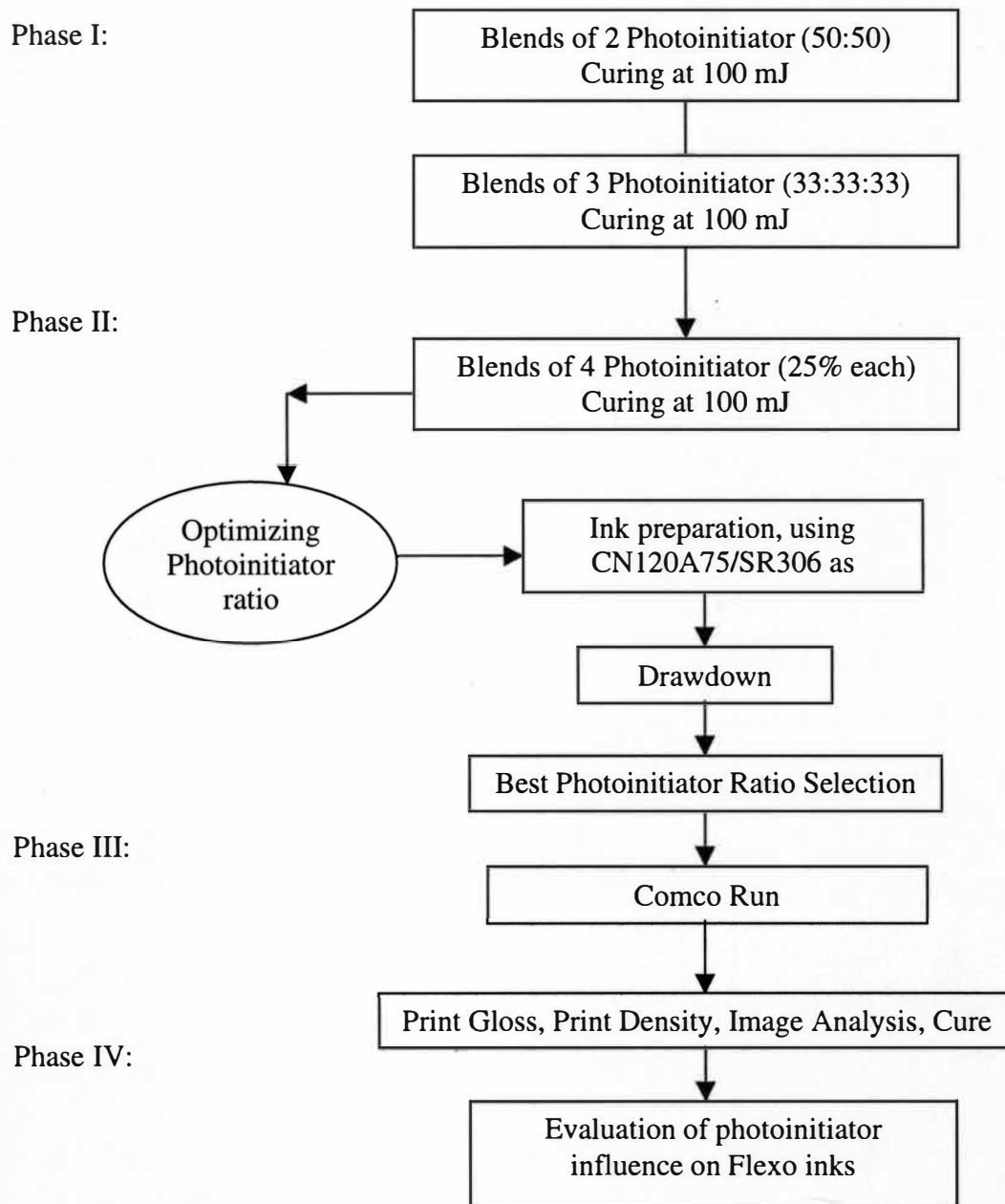


Figure 20: The Overall Experimental Plan Divided into the Four Phases

CHAPTER 5

EXPERIMENTAL

The energy of cure was determined using a Con-Trol-Cure® compact Radiometer # M007-008. Stain densities were measured with an X-Rite®418 densitometer. Absorption spectrum and extinction coefficient is measured by using an UV visual spectrophotometer.

All samples were cured using a curing unit containing an 'H' Hg arc lamp with a maximum output of 200 W and at a speed of 100 fpm.

Sample Preparation

In order to do this analysis, three sets of samples are prepared.

Sample Set #1

This set consists of all possible mixtures of photoinitiators mixed together two at a time on 50%-50% basis. These samples were melted together to check their solubility in one other. After melting them, standard solutions were prepared by using these melted blends and monomers CN120A75 and SR306.

Sample Set #2

This set consists of all possible mixtures of photoinitiators from sample set #1 cured at $100\text{mJ}/\text{cm}^2$ of energy, mixed together, three at a time in equal amount (33.3% of each).

Sample Set #3

This set consists of all possible mixtures of photoinitiators from sample set #2 cured at $100\text{mJ}/\text{cm}^2$ of energy, mixed together, four at a time.

Composition of Coatings

CN120A75: 50 parts.

SR306: 45parts.

Photoinitiator Blend: 7 parts.

These solutions were used to prepare draw -downs in order to measure degree of curing. KMnO_4 yellow stain density test was used to calculate the degree of curing.

KMnO_4 Yellow Stain Density Test

Yellow density left by 5 minutes exposure of KMnO_4 is measured for different blends of photoinitiators. To measure the degree of curing, draw-downs of standard solutions are analyzed. By plotting the graphs between cure energy and KMnO_4 stain density, differences in cure response can be detected. The ease of compounding of each of the photoinitiators is being monitored for each photoinitiator solution. For this

test, put a drop of KMnO_4 onto the coating and the wipe it off after 5 minutes. The yellow density of the stain will give the measure of curing. Higher the yellow density, lower will be the cure. It would not give any yellow stain if the film were not cured, as possibly KMnO_4 will not be oxidized by the oxygen. If there is no compound in coating to react with KMnO_4 or it will give the highest possible density if it is oxidized by the chemicals in the coating. Then the magenta and cyan density will play an important role.

Analysis

All samples were melted to see their nature and phase behavior at 100°C and at room temperature. They were also checked after a few days under normal room conditions. The reactivity of different photoinitiators was compared in monomer/oligomer solutions. The curing characteristics for each photoinitiator were monitored by examining test samples cured at a belt speed of 100 fpm and 200 watts of Hg lamp. Level of curing of photoinitiator, their appearance after a long period of time (20-25 days) under room conditions and their nature in one another is given in Appendix A, Table 31 and Table 32. All blends which are not chemically compatible (based on their chemical structures and cure analysis) will be discarded. Further testing will be done with different composition of photoinitiators (based on the overall reactivity of particular photoinitiator in the blend). The reactivity of different photoinitiators was compared in monomer/oligomer solutions.

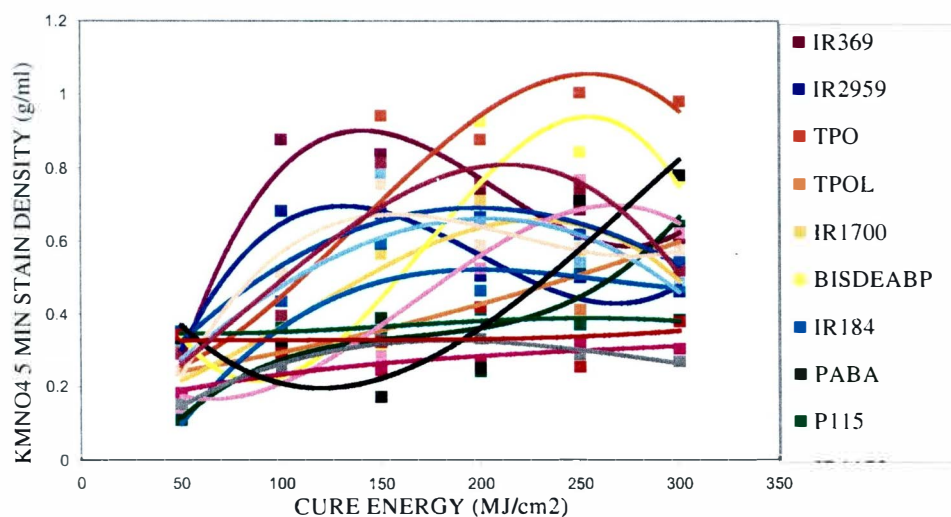


Figure 21. Yellow Stain Density Vs Cure Energy for the Blends of IR1700 and other PI Compounds

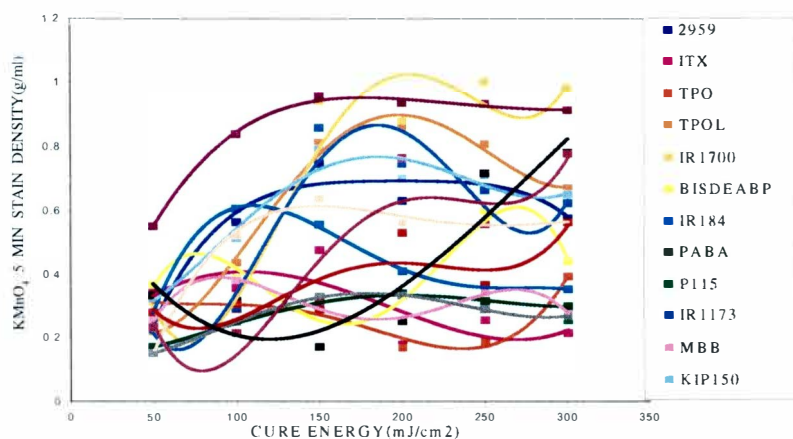


Figure 22. Yellow Stain Density Vs Cure Energy for the Blends of TPO and other PI Compounds

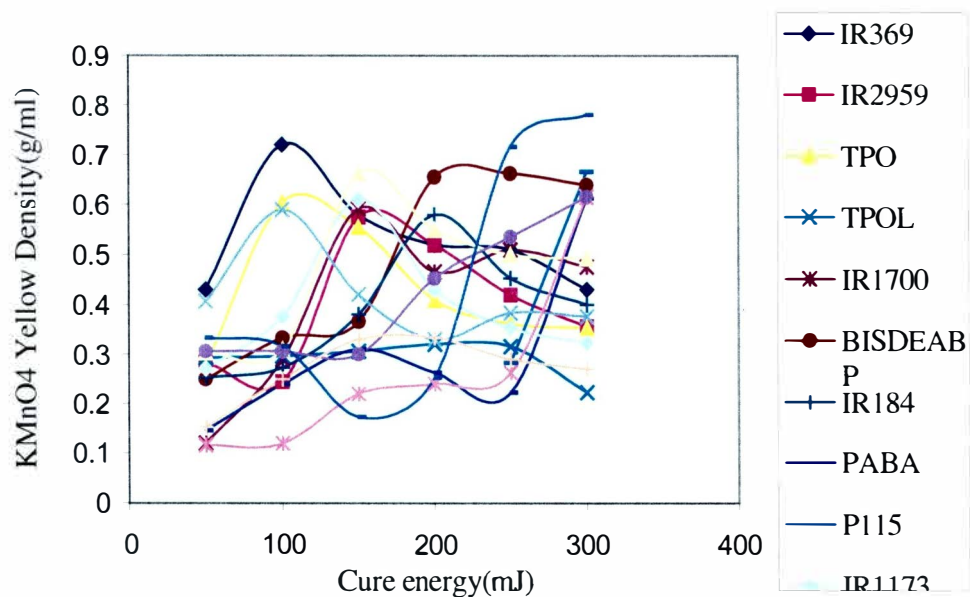


Figure 23. Yellow Stain Density Vs Cure Energy for the Blends of IR184 and other PI Compounds

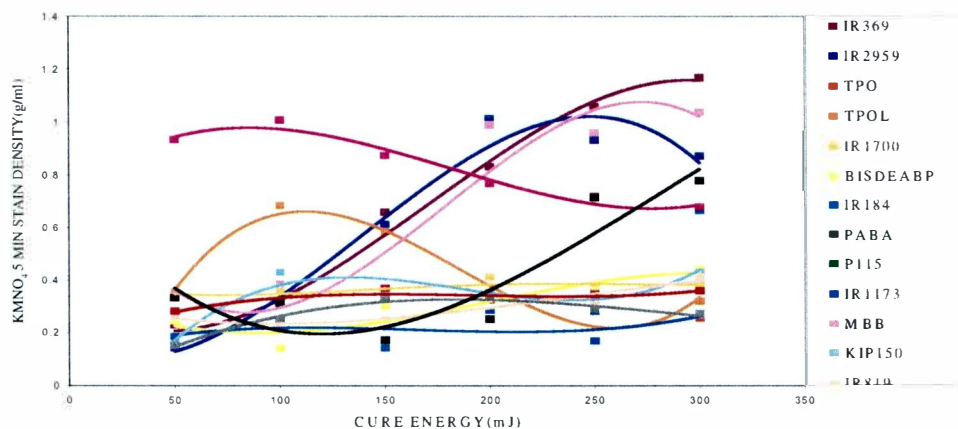


Figure 24. Yellow Stain Density Vs Cure Energy for the Blends of P115 and other PI Compounds

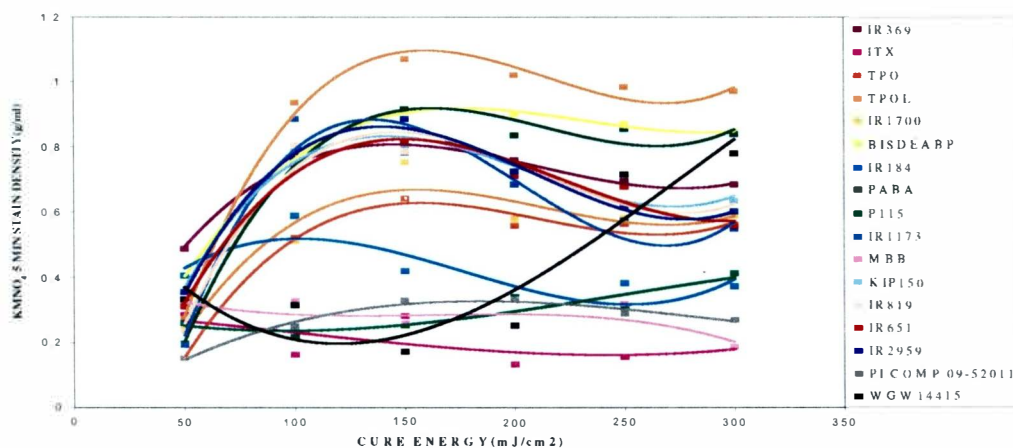


Figure 25. Yellow Stain Density Vs Cure Energy for the Blends of KIP and other PI Compounds

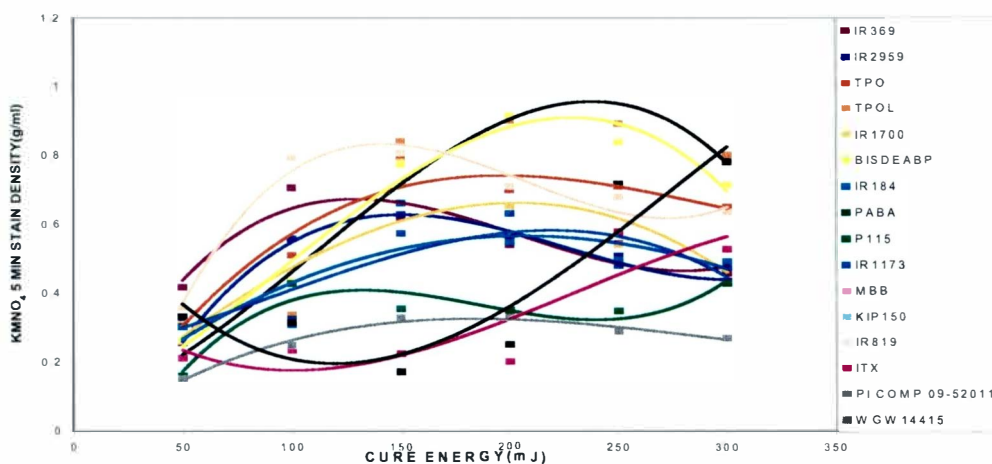


Figure 26. Yellow Stain Density Vs Cure Energy for the Blends of IR819 and other PI Compounds

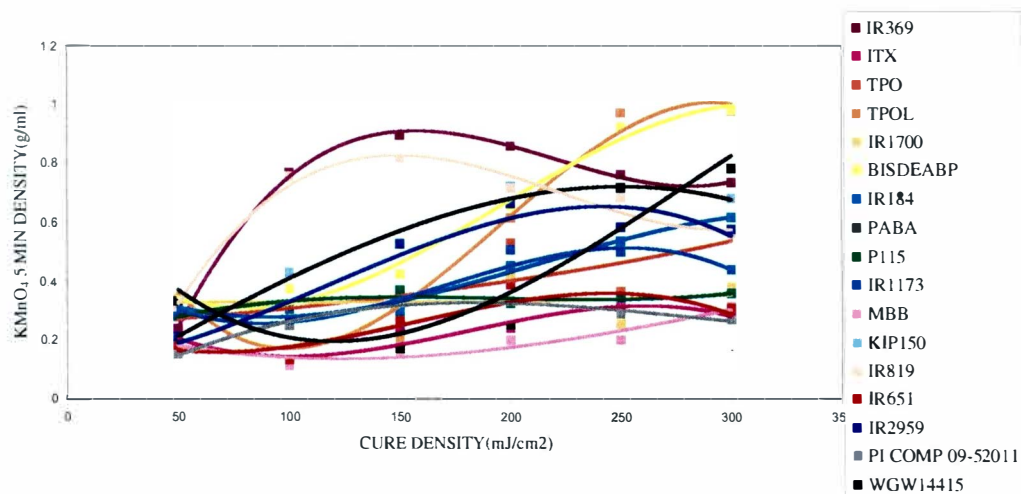


Figure 27. Yellow Stain Density Vs Cure Energy for the Blends of IR651 and other PI Compounds

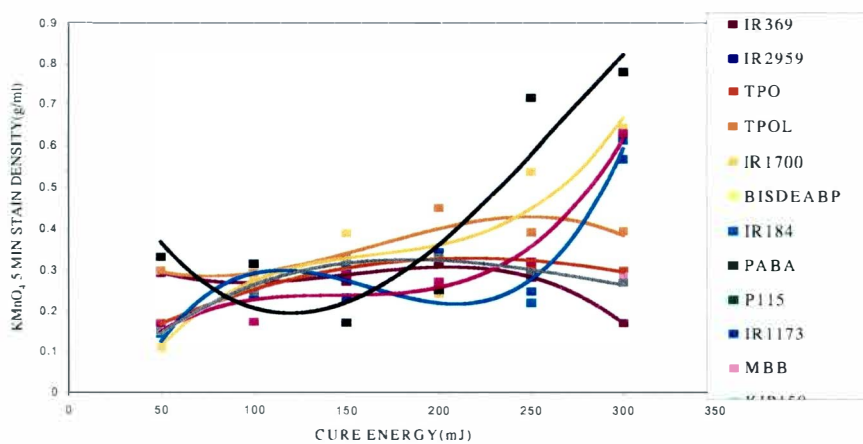


Figure 28. Yellow Stain Density Vs Cure Energy for the bBends of PABA and other PI Compounds

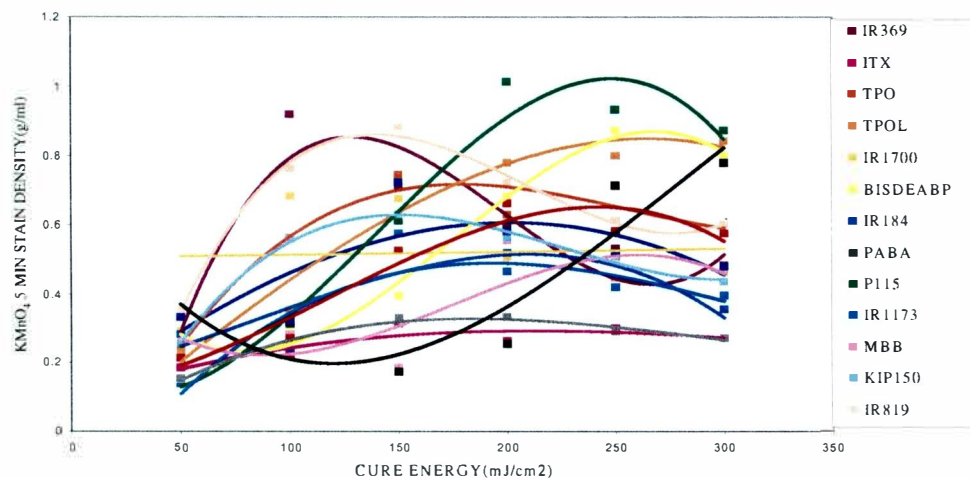


Figure 29. Yellow Stain Density Vs Cure Energy for the Blends of IR369 and other PI Compounds

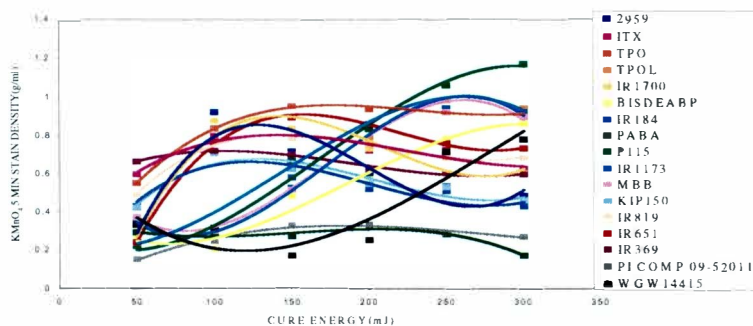


Figure 30. Yellow Stain Density Vs Cure Energy for the Blends of IR2959 and other PI compounds

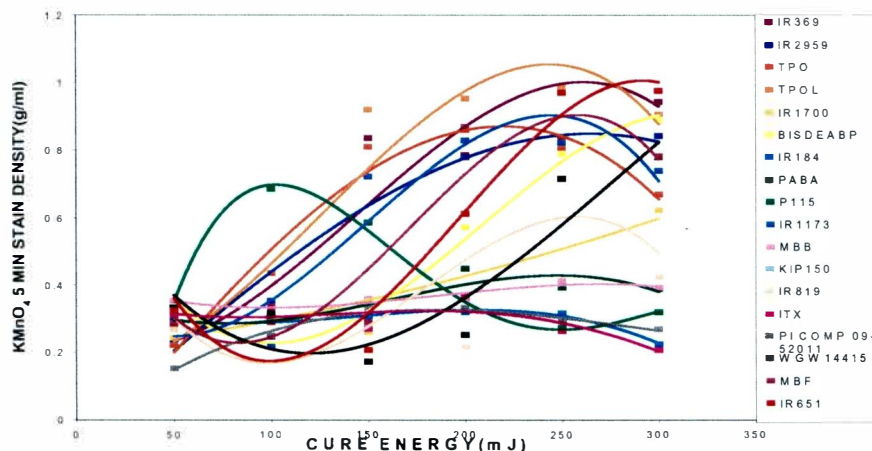


Figure 31. Yellow Stain Density Vs Cure Energy for the Blends of ITX and other PI Compounds

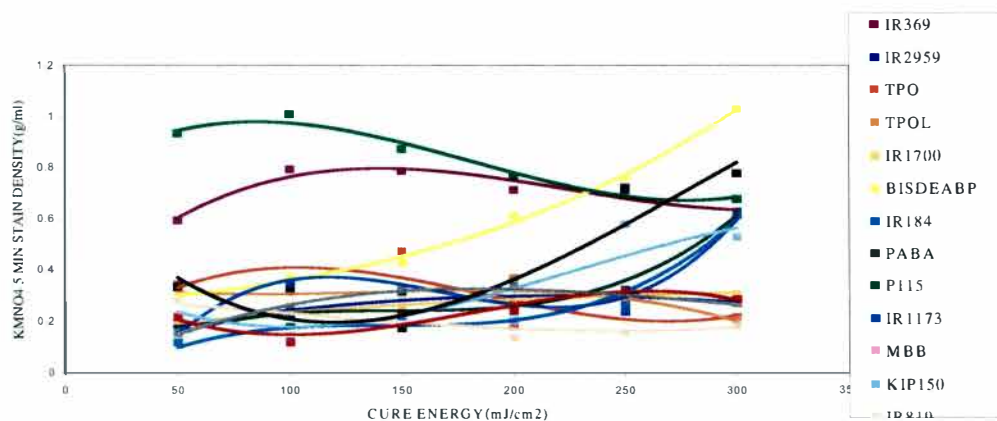


Figure 32. Yellow Stain Density Vs Cure Energy for the Blends of TPOL and other PI Compounds

Curing curves of the Standard Blends, Cured at 100 mJ (From Sample set #1)

Yellow Stain Density at different energy levels for standard photoinitiator blends of Flint inks were shown in Appendix A, Table 33.

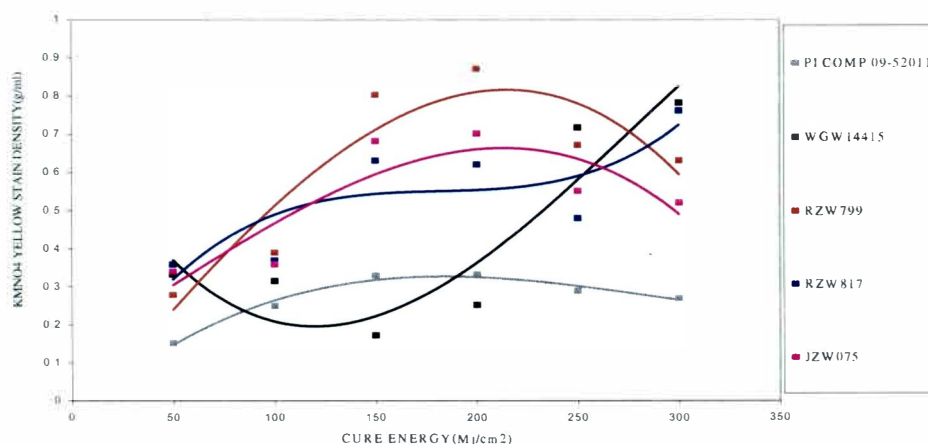


Figure 33. Yellow Stain Density Vs Cure Energy for the Standard Blends of Flint inks

Curing curves of the test blends, cured at 100 mJ (From Sample set #1)

Yellow Stain Density at different energy levels for test blends from sample set #1 were shown in Appendix A, table 34, 35 & 36. From table #34,35&36(Ref: Appendix A), the compounds were chosen, which cured at 100 mJ, to make sample set #2. Plotting the graphs between the cure speed and yellow stain densities monitored the curing characteristic for each blend. Yellow Stain Densities at different energy levels were shown in Appendix A, table 37, 38 & 39. More studies are required to decide the exact proportion of each photoinitiator in the blend. IR369 is a blend with strong absorption, at longer wavelength in the UV region, and with a tail in the visible region of light; therefore, it can be very effective in curing blue and black inks. It can be used in combination of a sensitizer like ITX. IR 369 also offers the additional benefit of being a low odor and low volatility photoinitiator.

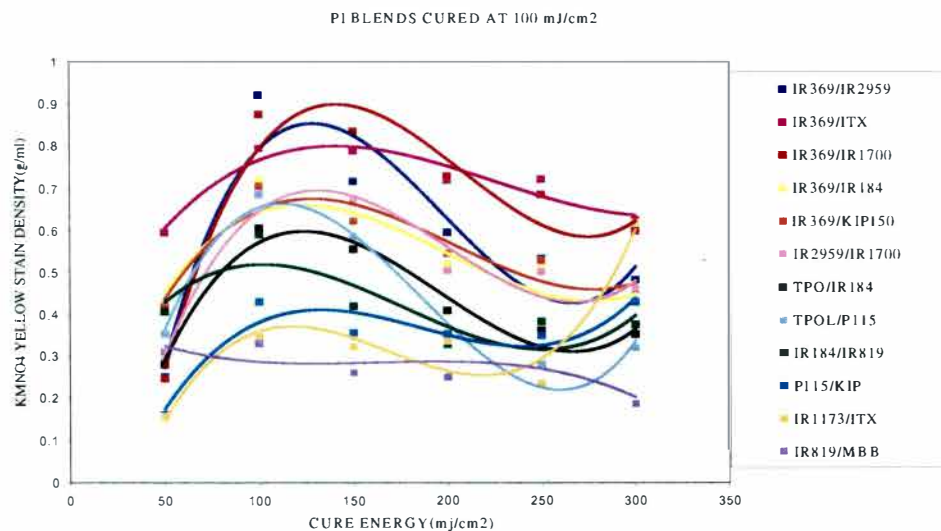


Figure 34. Yellow Stain Density Vs Cure Energy for the Blends of 2 PI Compounds Curing at 100 mJ

Curing curves of the blends, cured at 100 mJ (From Sample set #2)

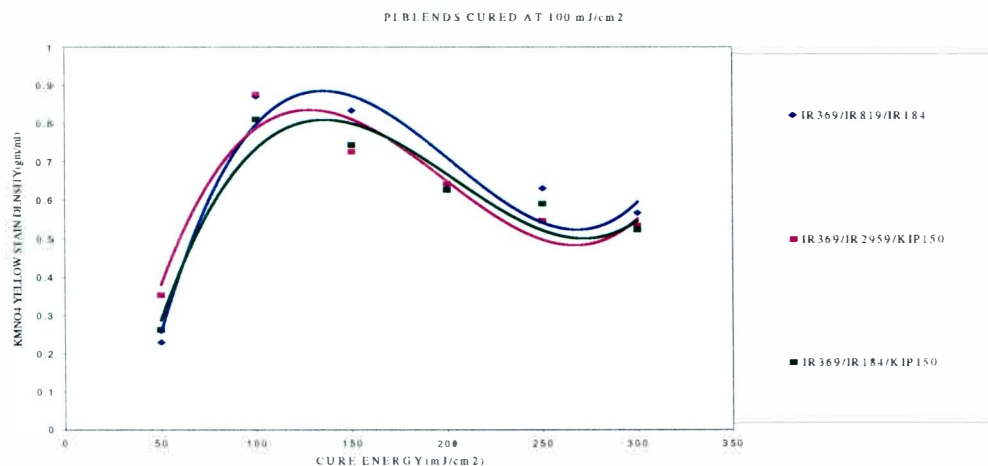


Figure 35. Yellow Stain Density Vs Cure Energy for the Blends of 3 PI Compounds Curing at 100 mJ

Curing curves of the blends, cured at 150 mJ (From Sample set #2)

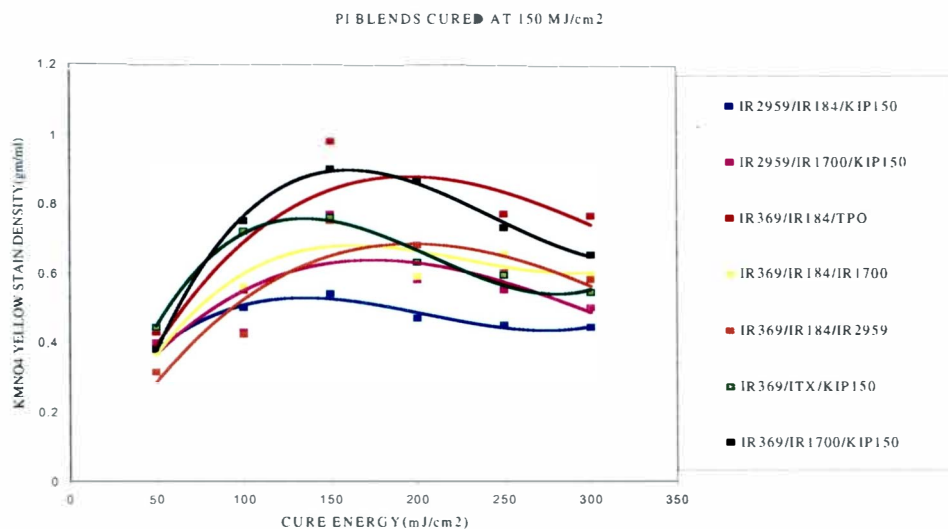


Figure 36. Yellow Stain Density Vs Cure Energy for The Blends of 3 PI Compounds Curing at 150 mJ

The results of sample set #2 are shown below in Table 19 & 20, The following combinations were obtained that cured at 100 mJ and 150 mJ.

Table19

Blends cured at 100 mJ/cm²

BLEND	UV ABSORPTION PEAKS, nm	APPEARANCE
IR369	325 –335	Crystallized
IR2959	275 –285, 320 –330	
KIP150		

Table 19 - Continued

BLEND	UV ABSORPTION PEAKS, nm	APPEARANCE
IR369 KIP150 IR184	325 –335 240 –250 , 320 –335	Highly viscous clear syrup
IR184 IR369 IR819	240 –250, 320 –335 325 –335 360 –365 , 405	Highly viscous clear syrup

Table 20

Blends cured at 150 mJ

BLEND	UV ABSORPTION PEAKS, nm	APPEARANCE
IR369 IR1700 IR184	325-335 245-325 240-250	Liquid
IR 369 IR1700 KIP150	325 –335 245 ,325	Crystallized
IR2959 IR184 KIP150	275 –285 240 –250 , 320 –330	Highly viscous clear syrup

Table 20 – Continued

BLEND	UV ABSORPTION PEAKS, nm	APPEARANCE
IR 369	325 –335	Highly viscous clear syrup
IR 184	320 –335 , 240 –250	
TPO		
ITX	325 -335	Highly viscous clear syrup
IR369		
KIP150		

These results reveal that blend of any of these could be the solution of this particular problem. More studies are required to decide the exact proportion of each photoinitiator in the blend. IR369 is a blend with strong absorption, at longer wavelength in the UV region, and with a tail in the visible region of light; therefore, it can be very effective in curing blue and black inks. It can be used in combination of a sensitizer like ITX. IR 369 also offers the additional benefit of being a low odor and low volatility photoinitiator. IR819 can be used for white, pastel; light colored inks due to its long wavelength absorption in combination with efficient photo bleaching and the possibility to form four reactive radicals. Blend of IR819 and IR184 can be used for highly pigmented systems. For coatings, products of alcohohydroxyketone (IR184, IR2959, IR1173) are preferred for curing un-pigmented, clear coatings and for delivering surface curing in high pigmented systems. IR819 is preferred for through curing.

current blends we have now, these combinations are curing much faster than the standard solutions (Ref: Appendix A, Table 33).

Table 21

Comparison of Final Results (test blends of three) with Standard Blends

STANDARD	TEST BLEND	ODOR	CURE ENERGY	STABILITY
WGW14415	-	Standard	600 mJ	Clear viscous syrup
RZW799	-	Standard	200 mJ	Clear liquid
RZW817	-	Standard	150 mJ	Clear liquid
PI Comp	-	Standard	150mJ	Clear viscous syrup
JZW075	-	Standard	200 mJ	Clear liquid
	ITX / IR369/KIP	Lesser than std.	150 mJ	viscous clear syrup
	IR2959/IR184/KIP	Very less than std.	150 mJ	viscous clear syrup
	IR369/IR1700/IR184	Lesser than std.	150mJ	Liquid with crystals
	IR184/IR369/IR819	Lesser than std	100 mJ	viscous clear syrup
	IR819/IR184/IR1173	Lesser than std.	150 mJ	Clear Liquid
	IR369/KIP/IR184	Lesser than std.	100 mJ	viscous clear syrup
	IR369/IR184/TPO	Very less than std.	150 mJ	viscous clear syrup

Extinction Coefficients using MeOH/CN120A75 as a background

UV absorption peaks of different photoinitiator compounds given by CIBA at concentration of 4×10^{-4} g/ml of MeOH. Typically, the referenced λ_{\max} in the UV spectrum for photoinitiators is the $\pi \rightarrow \pi^*$ transition in allowed photoinitiator process.

Extinction Coefficients using MeOH/CN120A75 as a background

UV absorption peaks of different photoinitiator compounds given by CIBA at concentration of 4×10^{-4} g/ml of MeOH. Typically, the referenced λ_{\max} in the UV spectrum for photoinitiators is the $\pi \rightarrow \pi^*$ transition in allowed photoinitiator process.

The $N \rightarrow \pi^*$ transition is the photochemical process attributed to Norrish type I photochemical reactions (*March: Advanced Organic Chemistry). To characterize the $N \rightarrow \pi^*$ extinction coefficient, I increased the concentration from 4×10^{-4} g/ml to 0.05g/ml. I used MeOH and CN120/TRPGDA as background end solvents for measuring the extinction coefficients.

Results

Table 22

λ_{\max} for Different Photoinitiator Compounds

	$\lambda_{\max} \pi \rightarrow \pi^* \text{ MeOH}$	$\lambda_{\max} n \rightarrow \pi^* \text{ MeOH}$	$\lambda_{\max} n \rightarrow \pi^*$ CN120/TRPGDA
Benzophenone	250	332	340
Irgacure 2959	275	320-340	320-340
Irgacure 907	310	320-325	320-340
Bis-DEABP	378	320-440	320-420
TPO	240	350-400	366, 380, 400

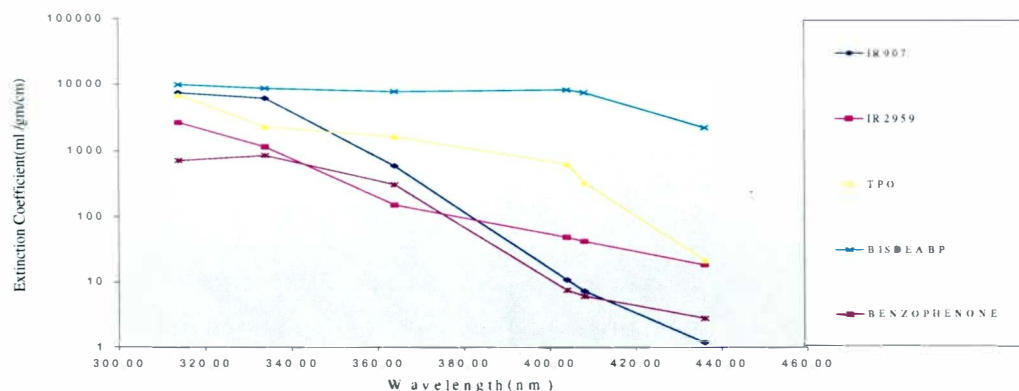


Figure 37. Mercury Vapor Lamp Compatability(MeOH as a Background)

Table 23

Extinction Coefficient(ml/gm/cm)

PI Compound	314 nm		364 nm	
	MeOH	CN120A75/SR306	MeOH	CN120A75/SR306
IR 184	4.35E+02	2.7716E+02	8.86E+01	5.485E+01
IR369	4.85E+04	1.547E+06	7.83E+03	2.231E+05
IR651	7.22E+02	6.348E+02	3.61E+02	5.016E+02
IR907	5.64E+04	1.1455E+04	4.67E+02	4.975E+02
IR1700	4.16E+03	4.014E+03	8.35E+02	3.482E+02
IR2959	2.57E+03	2.196E+03	4.88E+01	1.066E+02

This shift in the extinction coefficient can be explained based on polarity of background material, dipole-dipole interaction and hydrogen bonding. This change in

extinction coefficient is also depends on hydrophilic or hydrophobic nature of individual photoinitiator. Therefore for optimizing the effectiveness of photoinitiators in acrylated systems, we should:

1. Consider $N \rightarrow \pi^*$ extinction coefficient and λ_{\max} .
2. Use acrylated materials as the background.

Blend of four PI compounds 25% each were made. Based on yellow stain density and the cure analysis, it was found out that IR369, IR2959, IR184 and IR 1850 provide best through cure, surface cure with the absorption of lesser amount of energy. The blend of these four PI compounds works well for clear as well as for pigmented systems.

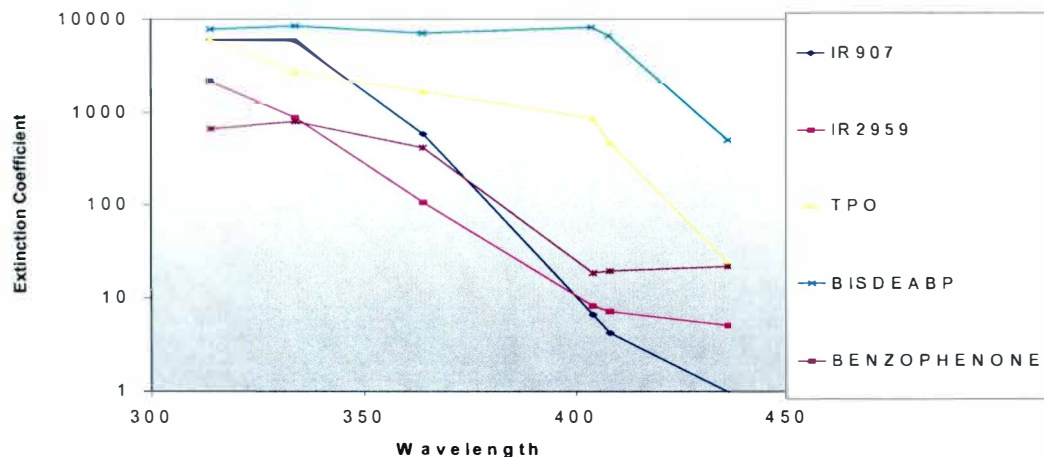


Figure 38. Mercury Vapor Lamp Compatibility (CN120A75/ Sr306 background)

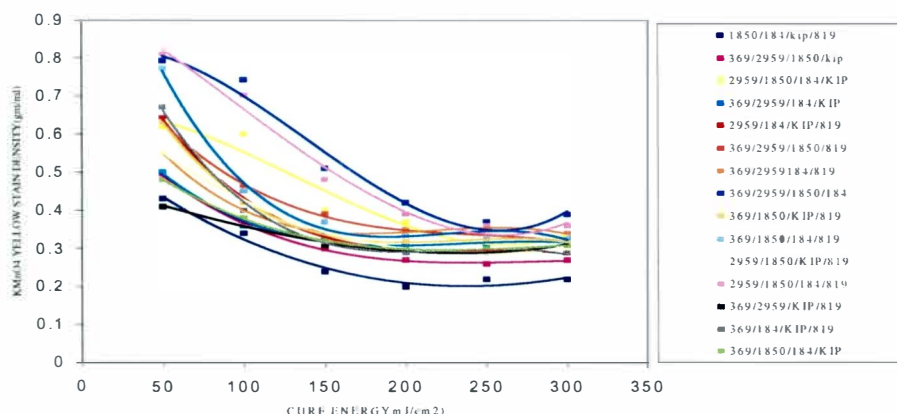


Figure 39. Blend of Four PI Compounds

Ink Cure Analysis

Indices 1 is related to O₂ Inhibition. Based on the blends of four: (a) KIP and IR2959 shows sensitivity to oxygen; and (b) IR369, IR184 and IR1850 show little sensitivity to oxygen. Based on the blends of three: (a) KIP and IR2959 shows sensitivity to oxygen; (b) IR369, IR184 and IR1850 show little sensitivity to oxygen.

Indices 2 related to Surface Cure. Based on the blends of four: (a) KIP and IR369 give high surface cure as compared to IR2959, IR1850, IR184, IR819 and ITX; (b) Rests of the photoinitiator are in the order: IR2959>IR1850> IR184> IR819 >ITX. Based on the blends of three: (a) IR369 give high surface cure as compared to IR2959, IR1850, IR184, IR819, TPO, MBB and ITX; (b) IR1850 and IR2959 have comparable surface cure properties.

Indices 3 Related to Cross-Link Density. Based on the blends of four: (a) IR369 give high cross-link density as compared to IR2959, IR1850, IR184, IR819 and ITX; (b) Rests of the photoinitiator are in the order: IR1850> IR184>

Indices 3 Related to Cross-Link Density. Based on the blends of four: (a) IR369 give high cross-link density as compared to IR2959, IR1850, IR184, IR819 and ITX; (b) Rests of the photoinitiator are in the order: IR1850> IR184> IR2959>ITX>KIP>IR819. Based on the blends of three: (a) IR1850 and IR2959 have comparable cross-link density; (b) Rests of the photoinitiator are in the order: IR1850= IR2959>KIP>IR819>ITX>TPO>MBB.

Indices 4 is Related to Through Cure. Based on the blends of four: (a) IR184 give high through cure as compared to IR369, IR2959, IR1850, KIP, IR819 and ITX; (b) Rest of the photoinitiators are in the order : IR 369> IR 2959 >ITX> IR 184> MBB> TPO> IR819> KIP.

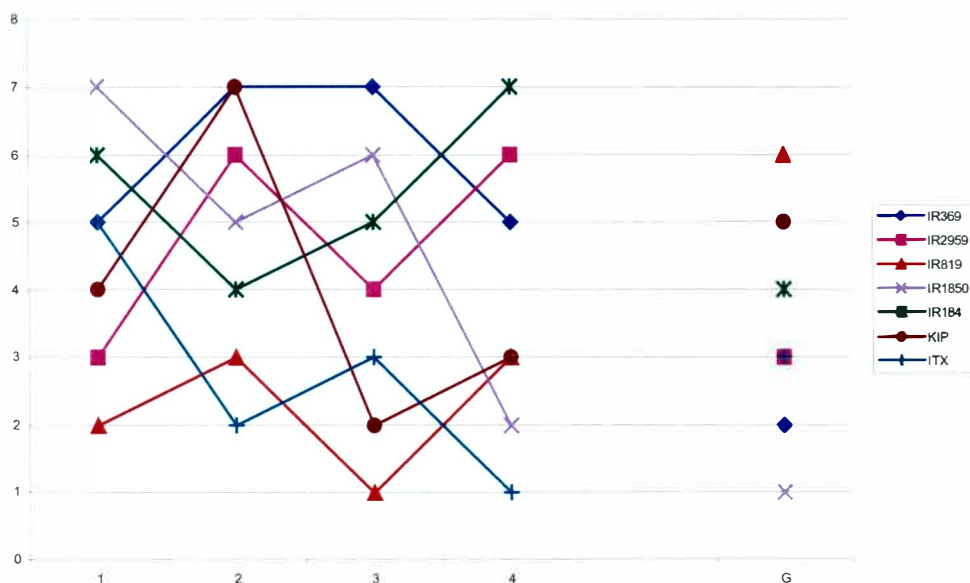


Figure 40. ICA and Gloss

Here in the graph, the x-axis represents the four indices and Y-axis represents the gyger count values of the radioactive material. The count value is time dependent.

not cured. It did not take anytime for radioactive material to penetrate into, as there were no crosslinking at all.

The data of this graph is shown in Appendix A, Table 42.

Trial Run

Based on the results one can easily decide that which photoinitiator is good for which type of curing. In addition, a combination of these photoinitiators can also provide us the best of all properties together in one blend. Results indicate that IR369, IR184, IR2959 and IR1850 give high surface as well as through cure.

Now to find out the best composition of these Photoinitiators into the blend, (so that we can get all desired properties along with minimum cost) several trials were done based on molar ratios. As we know, that curing depends on the number of free radicals available in the blend to absorb the energy and do cross-linking. Therefore, by calculating the no of moles going into the system, we can actually optimize the ratio of photoinitiators into the particular blend.

Therefore, In the blend of four PI compounds (25% each):

No of moles of IR 369 in 25g	$= (6.023 \times 10^{23} / 366.6) \times 25$	$= 4.107 \times 10^{22}$ moles
No of moles of IR 2959 in 25g	$= (6.023 \times 10^{23} / 224.26) \times 25$	$= 6.17 \times 10^{22}$ moles
No of moles of IR 1850 in 25g	$= (6.023 \times 10^{23} / 288.5) \times 25$	$= 5.219 \times 10^{22}$ moles
No of moles of IR 184 in 25g	$= (6.023 \times 10^{23} / 204.27) \times 25$	$= 7.37 \times 10^{22}$ moles

Molar ratio:

IR369: IR2959: IR1850: IR 184 :: 1: 1.5 : 1.3 : 1.79

Based on some facts like IR369 gives odor problems and it is not stable (ref: chemical structure) also and with the help of molar ratio, we can reduce the no of moles of IR369 in the blend. IR184 gives the best surface cure as well as through cure. Also IR184 gives the maximum free radicals per gram of PI compound.

Therefore by increasing the ratio of IR 184, we can achieve good results with stability and less odor (ref: previous experiments results and graphs shows that IR184 is stable, easily cured, non odorous compound works well for pigmented systems). IR 2959 works well with the combination of IR369 and IR1850 (ref: chemical structure). Therefore by using these facts along with hit and trial method, following molar combination were made and tested for surface cure and through cure for both systems clear as well as pigmented (Black).

Table 24

Molar Ratio for Different Ink Samples

Sample No	IR369	IR2959	IR1850	IR184
1	1	1.5	1.3	3.6
2	1	2.5	1.3	4.8
3	1	3.0	2.1	3.6
4	1	2.5	2.6	3.6
5	1	2.0	2.6	4.2
6	1	4.5	1.7	3.0
7	1	4.0	3.9	5.4
8	1	4.0	4.5	4.8
9	1	5.3	2.6	6.3
10	1	4.5	3.3	6.3

These samples of photoinitiators were mixed with the standard black base composed of CN120A75, SR306 and black pigment

Pigment: monomer :: 12.5: 87.5

CN120A75: SR306 :: 50: 45

Ink: photoinitiator :: 10:1

Results Based on Tape Test and Alcohol Test

Tape test is based on the amount of ink came out with the tape and alcohol test is the measurement of number of strokes required to remove the ink from the surface. These tests can be confirmed by measuring the black density before and after the tape test and alcohol test.

Table 25

Result of Tape Test and Alcohol test for test inks

Sample No	Molar Ratio	Tape Test	Alcohol Test
1	1 : 1.5 : 1.3 : 3.6	Pass	5-6 strokes
2	1: 2.5 : 1.3: 4.8	Pass	8-10 strokes
3	1: 3.0 : 2.1 : 3.6	Pass	6-8 strokes
4	1 : 2.5 : 2.6 : 3.6	Pass	7-8 strokes
5	1 : 2.0 : 2.6: 4.2	Pass	8-10strokes
6	1 : 4.5 : 1.7 : 3.0	Pass	5-6 strokes
7	1: 4.0 : 3.9 : 5.4	Pass	Pass(12-15 strokes)
8	1 : 4.0 : 4.5 : 4.8	Pass	8-10 strokes
9	1 : 5.3 : 2.6: 6.3	Pass	Pass(15-17 strokes)
10	1 : 4.5 : 3.3 : 6.3	Pass	Pass(15-17 strokes)

Based on these results, sample number 7, 9 and 10 were selected.

Table 26

Ink Information

Unit #/color	2
Ink	Black
Reducing monomer	CN120A75, SR306
Additional Photoinitiator	IR369, IR2959, IR1850, IR184
CN120A75: SR306	50:45
Base: Photoinitiator	10:1

Composition for 5.6 liters of black ink

CN120A75 = 2611 g

SR306 = 2297 g

Pigment = 700g

Trial runs were done on COMCO COMMANDER Press using the above base formulation and pigment blends. Substrate for trial run is 7.5 Uncoated pretreated V-Max opaque polyethylene.

Table 27

Press Information

STATION #	STATION 2
Anilox LPI	600

Table 27 – Continued

Anilox BCM	2.61
Plate Mat	EXL DUPONT
Sticky Back	Rotometric (12cm*12cm)

Web speed during run is 300 fpm and the power of UV lamps was 400 watts/inch.

Properties of Trial Samples After Printing

Table 28

Properties of Trial Samples

Sample #	Sample 7	Sample9	Sample 10
Print Density	1.13	1.04	1.05
Ink Viscosity (#4 spindle @ 60rpm, factor = 100)	30.6 cp.	30.9 cp.	30.6 cp.
Tape Test	Pass	Pass	Pass
Alcohol Test	Pass	Pass	Pass
Delta Gloss	35	33	32

Image Analysis

Table 29
Image Analysis

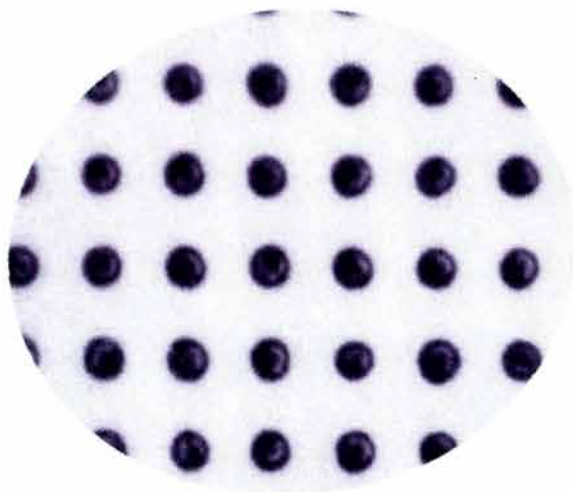
Sample	Area(microns ²)			
	Min	Max	Mean	Std dev.
# 7	11833.3	15844.4	13751.0	899.88
#9	12688.9	16844.4	14255.1	721.07
#10	11788.9	14577.8	13290.8	548.55

Sample	Perimeter(microns)			
	Min	Max	Mean	Std dev.
# 7	383.4	442.9	411.8	35.1
#9	297.7	463.3	421.8	27.9
#10	328.5	424.3	405.5	23.5

Sample	Roundness			
	Min	Max	Mean	Std dev.
# 7	1.0	1.1	1.0	0.002
#9	1.0	1.0	1.0	0.003
#10	1.0	1.8	1.2	0.209

*dev. = deviation

Sample #7



Sample #9

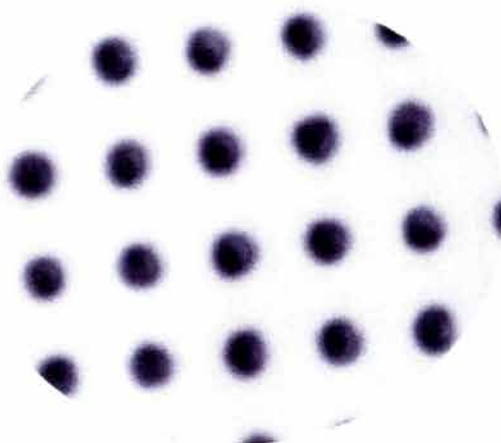


Figure 41: Image Analysis

Sample #10

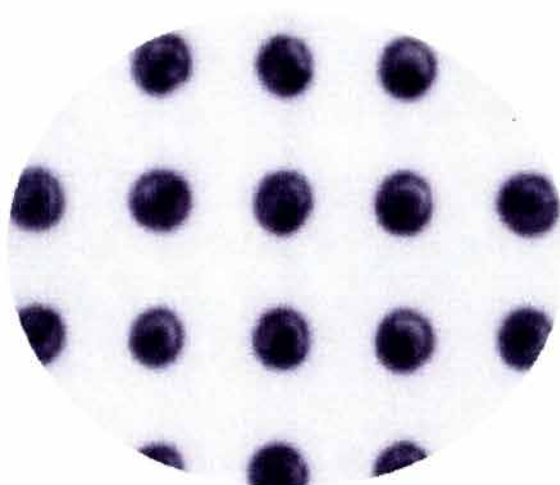


Figure 41 - Continued: Image Analysis

ICA Test Results and Discussion

Table 30

Ink Cure Analysis

No.	I	II	III	IV
Ink#7	197	88	220	718
Ink#9	185	88	154	577
Ink#10	194	87	143	576

Here: I, II, III, IV → indicate the gyger (molecular) count of radioactive material penetrating through the film. The graphs obtained on the ink cure analyzer

represents molecular counts of radioactive material Vs. time counts. In the first section (Index I), the index depicts the sensitivity towards oxygen called oxygen Inhibition. Higher counts means less sensitivity to oxygen. The ink #7 has higher count value for I indices in comparison to ink # 9 and ink # 10 means ink # 7 is less sensitive to oxygen.

The second index depicts surface cure. All counts after this point represent solvent, which has permeated the ink. If the count value is higher, it means that it takes more time to penetrate into, means more cured film. Count of #7 ink are comparable to the counts for # 10 and #9. It indicates that all samples have same surface cure.

The third index depicts crosslink density. Results indicate that crosslink density of ink #7 is much higher in comparison of ink #9 and ink #10 is higher in comparison of other inks. Therefore, ink #7 has more cross-link density.

The values of indices IV of ink#7, representing through cure are comparably higher than # 10 and #9, which indicate the ink #7 is curing faster in comparison of other two. Therefore, we can conclude that #7 ink is curing better in comparison of other inks.

CHAPTER VI

CONCLUSIONS

1. Three samples of inks with different photoinitiator molar ratio, (1:4.0:3.9:5.4), (1: 5.3: 2.6: 6.3) and (1: 4.5: 3.3: 6.3) using photoinitiator IR369, IR2959, IR184 and IR 1850, were produced. The ink viscosity for ink #7, ink #9 and ink #10 were 30.6cp, 30.9cp and 30.6cp respectively and the print density was 1.13, 1.04 and 1.05 respectively. Lower values of density indicate that the ink was not properly milled and the particle size was higher in the ink. Higher print density of ink #7 indicates higher linking, more compact print. (Ref Table 28)
2. Results of tape test and alcohol test were positive. It indicates that all ink samples were curable. (Ref Table 28)
3. The gloss of print samples was in the range of 45 to 50. Due to uneven distribution of particle size in inks, the gloss values are little lower but still they are in the acceptable range. The gloss values of ink #7 were higher in comparison of ink # 9 and ink # 10. It indicates that there was more crosslinking occurred in ink #7 means the curing rate of ink # 7 is higher than that of ink #9 and ink # 10.(Ref: Table 28)
4. The mean dot area for ink #7, ink #9 and ink # 10 was 9445.30, 9838.33 and 9609.044 respectively and the roundness of dots for ink #7, ink #9 and ink #10 was approximately 1.0. Dot area decreases with the increase in the cross-linking.

5. The lower values of dot area for ink # 7 as compared to ink #9 and ink # 10 indicates that there is more cross-linking occurred in ink # 7. (Ref Table 29)
6. The ink cure analysis of ink #7, ink #9 and ink #10 indicates that the surface cure and through cure of ink #7 were higher in comparison of ink # 9 and ink # 10.

High counts are interpreted as meaning that the ink is well cured and it takes more time to penetrate into the ink film. The counts for ink # 7 were higher in comparison of ink #9 and ink # 10. (Ref Table 30)

CHAPTER VII

RECOMMENDATIONS FOR FUTURE STUDY

Additional studies are required to better understand the influence of photoinitiator on pigmented as well clear systems. This includes the evaluation of the print quality in terms of image analysis and cure analysis, which will assist in predicting the print quality and degree of curing accurately. It would also be helpful to test these photoinitiator blends for different colors. It is also recommended to use the IR 184 with higher degree of surface cure and through cure in order to get better cross-linking and better curing. The use of IR 369 should be as minimum as possible as it cause problems like odor and swelling of plates.

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10. <http://www.fusionuv.com/apps/>

Appendix A

Here: S =solid, SS =semisolid, L =liquid, V = viscous, R =Recrystalized

Table31

Level of Curing of Photoinitiator And Their Nature in one Another

Energy mJ	IR369	IR2959	TPO	TPOL	IR1700	BISD
100	2959 (S)	IR369(S)	IR184(V)	P115(L)	IR369(R)	
100	ITX (R)	IR1700(V)	MBB(S)		IR2959(V)	
100	IR1700(R)				IR184(L)	IR819(S)
100	IR184 (R)				IR1173(L)	
100	KIP(S)				KIP150(V)	
150	TPO(S)	IR2959(S)	IR2959(S)	IR369(S)	IR819(S)	
150	TPOL(S)		ITX(S)		MBF(L)	
150	IR819(S)	IR819(R)	TPO(R)		TPOL(V)	IR1700(SS)
150	IR651(SS)	KIP(SS)	IR1173(L)		IR1700(L)	IR184(SS)
150		IR1173(SS)	KIP(S)		BISD SS)	KIP150(V)
150		IR184(R)	IR819(S)		IR651(L)	
150		TPO(S)	IR369(S)		P-115(L)	
200	PABA(S)	TPOL(S)	TPOL(R)	TPO(R)	TPO(V)	TPO(S)
200		P-115(S)	PABA(R)	IR1700(V)	MBB(R)	IR1173(SS)
200		MBB(R)	IR651(V)	IR184(L)	ITX(SS)	IR2959(SS)

Table 32

Level of Curing of Photoinitiator and Their Nature

Cure mJ	IR1173	MBB	KIP150	IR819	ITX	IR651
100	IR819(R)	TPO(S)	IR369®	IR184(V)	IR1173(L)	
100	ITX(L)		P-115(SS)	IR1173(V)	KIP(S)	
150	TPO(L)		TPO(R)	ITX(R)	IR2959(V)	P115(S)
150	IR1700(L)		IR1700(V)	IR651(R)	TPO(R)	IR819(V)
150	IR184(L)		IR184(L)	TPOL(SS)	P-115(V)	
150			IR819(V)	IR1700(L)	IR819(SS)	
200	TPOL(V)	IR2959(V)	BISD(S)		TPOL(S)	TPO(S)
200	P115(S)		IR1173(V)			IR1700(L)
200	IR1173(L)					IR2959(V)
200	MBB(L)					IR1173(L)
200	KIP(V)					IR651(SS)
200	IR651(R)					
250	IR369(R)	IR369(SS)	ITX(L)		IR1700(V)	ITX(S)
250	BISD(S)	TPOL(S)			IR651(S)	
250		IR1700(S)				
OVER 300	WGW(V)	IR819(V)		P115(S)	BISD(S)	TPOL(SS)

Table 33

Yellow Stain Density at Different Energy Levels for Standard PI Blends

Cure Energy	PI COMP 09-52011	WGW14415	RZW799	RZW817	JZW075
50	0.153	0.333	0.28	0.36	0.34
100	0.25	0.316	0.39	0.37	0.36
150	0.33	0.173	0.8	0.63	0.68
200	0.333	0.253	0.87	0.62	0.7
250	0.29	0.716	0.67	0.48	0.55
300	0.27	0.78	0.63	0.76	0.52

Table 34

Yellow Stain Density at Different Energy Levels For The Blends
of 2 PI Compounds (50:50)

Cure energy	IR369/IR2959	IR369/ITX	IR369/IR1700	IR369/IR184
50	0.25	0.596	0.246	0.43
100	0.92	0.796	0.876	0.72
150	0.716	0.79	0.836	0.58
200	0.596	0.72	0.73	0.52
250	0.533	0.723	0.686	0.51
300	0.483	0.626	0.6	0.43

Table 35

Yellow Stain Density at Different Energy Levels For The Blends
of 2 PI Compounds

Cure energy	IR369/KIP150	IR2959/IR1700	TPO/IR184	TPOL/P115
50	0.42	0.29	0.28	0.353
100	0.706	0.683	0.606	0.686
150	0.623	0.676	0.556	0.586
200	0.546	0.506	0.41	0.326
250	0.53	0.503	0.363	0.28
300	0.46	0.463	0.353	0.32

Table 36

Yellow Stain Density at Different Energy Levels For The Blends
of PI Compounds

Cure energy	IR184/IR819	P115/KIP	IR1173/ITX	IR819/MBB
50	0.406	0.16	0.156	0.31
100	0.59	0.43	0.346	0.33
150	0.42	0.356	0.323	0.26
200	0.33	0.353	0.336	0.25
250	0.383	0.35	0.236	0.32
300	0.376	0.43	0.62	0.186

Table 37

Yellow Stain Density at Different Energy Levels For The Blends
of 3 PI Compounds (33.3:33.3:33.3)

Cure energy	IR369/IR819/IR184	IR369/IR2959/KIP150	IR369/IR184/KIP150
50	0.23	0.353	0.263
100	0.87	0.876	0.81
150	0.833	0.726	0.743
200	0.64	0.643	0.626
250	0.63	0.546	0.59
300	0.566	0.533	0.523

Table 38

Yellow Stain Density at different energy levels for the blends
of 3 PI compounds

Cure energy	IR369/IR184/ IR1700	IR369/IR184/ IR2959	IR369/ITX/ KIP150	IR369/IR170 0/KIP150
50	0.373	0.316	0.443	0.38
100	0.56	0.423	0.72	0.75
150	0.75	0.75	0.76	0.9
200	0.59	0.68	0.63	0.87
250	0.653	0.6	0.593	0.73
300	0.593	0.58	0.543	0.65

Table 39

Yellow Stain Density at Different Energy Levels for The Blends
of PI Compounds

Cure energy	IR2959/IR184/KI P150	IR2959/IR1700/KI P150	IR369/IR184/TPO
50	0.38	0.4	0.43
100	0.5	0.43	0.55
150	0.54	0.77	0.98
200	0.47	0.58	0.87
250	0.45	0.55	0.77
300	0.443	0.5	0.763

Table 40

Extinction Coefficients Using MeOH as a Background

PI COMPOUND	314Nm	334Nm	364Nm	404Nm	408Nm	436Nm
IR907	7543.3	6200.6	595.08	11.095	7.4578	1.2188
IR2959	2663.2	1152.82	151.638	49.144	42.454	18.7378
TPO	6855.8	2261.4	1617.96	628.72	325.76	21.826
BISDEABP	10030.8	8836.2	7904.00	8379.8	7597.4	2241.4
BENZOPHENONE	712.16	851.64	307.94	7.6942	6.2046	2.8438
IR 184	1485	NA	135.00			

Table 41

Extinction Coefficients Using CN120/TRPGDA as a Background

PI COMPOUND	314Nm	334Nm	364Nm	404 Nm	408 Nm	436 Nm
IR907	6125	5956.2	583.32	6.6184	4.242	0.9842
IR2959	2196	888.04	106.658	8.1958	7.1506	5.0792
TPO	6223.4	2706.2	1677.64	864.58	470.74	23.694
BISDEABP	7928	8621.4	7236.8	8317.6	6759.4	501.42
BENZPHENONE	674.26	794.34	415.64	18.4612	19.3862	21.718

Table 42

Results of Ink Cure Analysis and Gloss

INK	I	II	III	IV	GLOSS
369/1850/184	388	240	213	245	44.3
369/2959/1850	308	200	161	160	43.5
KIP/1850/184	193	141	90	73	40.1
KIP/ITX/1850	121	98	50	183	25.9
184/369/819	292	196	150	149	36.9
369/2959/ITX	196	140	110	210	34.6
369/2959/KIP	357	229	196	169	38.7
369/184/KIP	287	198	154	116	42.9
KIP/2959/1850	134	113	76	2	37.03

Table 42 - Continued

INK	I	II	III	IV	GLOSS
369/ITX/184	191	139	102	124	35.4
KIP/ITX/369	166	126	82	181	33.3
KIP/2959/184	308	194	150	48	34.8
369/ITX/1850	149	115	76	17	36.2
819/184/MBB	29	50	13	105	33.9
369/1850/KIP	202	147	99	117	39.4
184/369/TPO	194	139	101	159	41.2
369/2959/184	306	206	156	178	40.8
184/TPO/819	252	163	127	99	40.4
KIP/ITX/184	170	121	95		33.1

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