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THE EFFECTS OF ELECTRON BEAM CURED COATINGS ON POLYMER SUBSTRATES

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Susan Selke

Major Professor's Signature

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THE EFFECTS OF ELECTRON BEAM CURED COATINGS ON POLYMER SUBSTRATES

By

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ABSTRACT

THE EFFECTS OF ELECTRON BEAM CURED COATINGS ON POLYMER SUBSTRATES

By

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Like ultraviolet curing, electron beam curing is a growing technology which is now commercially available. The electron beam curing technology has been proved to offer numerous benefits and advantages in every aspect of its application. Various industries have been switching to the technology in order to improve performance, increase profitability and gain environmental acceptance. However, in the flexible packaging materials industry, converters and end users currently appear to be fragmented in their approach to this technology. Because concerns have been expressed about the effect of e-beam curing on the functionality of flexible packaging materials, this study will provide converters and end users with information that will facilitate their decision about whether e-beam cured coatings are appropriate for their applications. Performance of two co-extruded and two metallized films coated with electron beam curable coatings were studied to determine whether any changes occurred as a result of the electron beam-cured coatings. Tests of selected mechanical properties and migrational behavior of the substrates were done on both the base films (uncoated and uncured) and the treated films (coated and cured) for comparison.

For my beloved husband and daughter, Azril Haris and Nur Alya Batrisyia

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TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

INTRODUCTION

The use of electron beam (EB) curable coatings, inks and adhesives has increased dramatically over the last decade. Energy curing use in packaging decoration and protection started with the first commercial run of UV (ultraviolet light) curing inks and coatings in 1969.(10,12) The use of EB products for similar application took another ten years for commercialization due to economic considerations. These technology are used in a variety of applications such as printing inks, overprint varnishes, release coatings, primers, pigmented paints, clear topcoats, pressure sensitive adhesive and the list of applications keeps growing within a very wide range of industries.(50) Flexible films used as packaging materials is one of the industries that are developing the use of EB curing technology in many applications.

The recent advances in electron beam curing chemistry and curing equipment encouraged the increased use of this technology, besides offering numerous advantages over conventional curing systems. RadTech International summarized three compelling reasons to convert to UV and EB technology.(42) The first, improved productivity, is based on the speed of curing, which is generally less than a second, compared to conventional coating methods. Compared to water base coatings web line speeds of 500 feet per minute (15), it is not uncommon for EB or UV technology to have web line speeds of 1,000 feet per minute. The second advantage is suitability for sensitive substrates because most EB ad UV systems do not contain water or solvent. With total control of the cure temperature, the process becomes a very practical choice for heat-sensitive substrates. The third reason is that it is both environmentally and user friendly. Since typical compositions are solvent-free, emissions and flammability are no longer an issue. Compatibility with virtually any application technique is a characteristic with light cure systems and space requirements are minimal. For example, UV lamps can generally be installed on existing production lines. More advantages of this technology will be explained in detail in the literature review.

Despite all of these advantages, UV or EB still have not been universally accepted as technologies of choice for converters. The reasons seem to be most evident in flexible packaging, particularly for food packaging. The three major reasons are the cost of materials, the fear of adopting a relatively new and radical method, and the impression that that the chemistry is or can be harmful.(12,24) Actually all these reasons unfounded. A review of the literature shows that application of this technology will result in significant savings on materials and equipment costs, and that they are safer than conventional systems. (12, 17, 21, 38, 43)

Another concern of converters and end users regards the actual application of EB curing to flexible packaging materials as the substrate. The impact of the EB curing on the substrate itself needs to be studied, since the energy can cause changes within the substrates just as the energy will cause the ink or coating to crosslink. As EB energy can be used to alter products to achieve desired features, further exposure might result in degradation of those properties. Based on these concerns, this study investigated the effect of EB curing on the functionality of several flexible packaging materials. The effects of EB curing of overprint varnishes on different laminating films were quantified. Selected mechanical and chemical properties of substrates were evaluated to determine what changes occurred as a result of the e-beam cured coatings. The results of this study will provide converters and end users with information that will facilitate their decision about whether EB cured coatings are appropriate for their applications.

CHAPTER 1

LITERATURE REVIEW

1.1 Electron beam source and radiation

Electron beams are generated using electron guns in a manner similar to that used in a TV picture tube. A beam from these guns can be deflected and focused magnetically to create a small spot that moves rapidly. Another way to generate EB is by using linear filaments and cathodes, directed by electrostatic electrodes to form their image on a substrate, as in Figure 1. For higher energy and higher production speed requirements, multiple filaments or cathodes can be used.

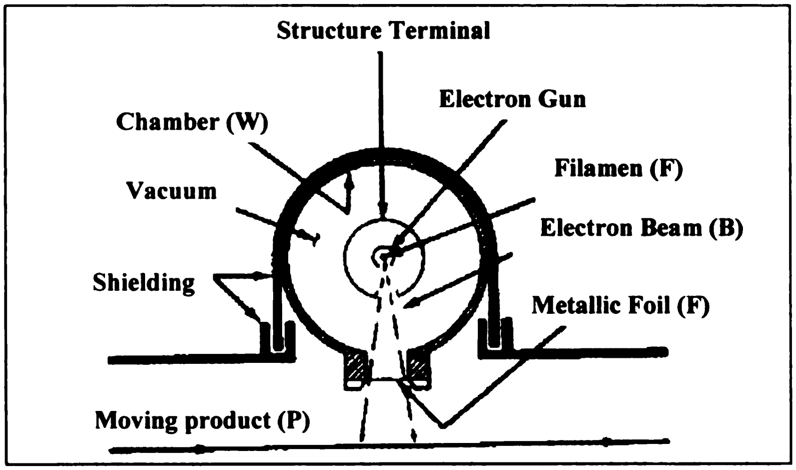


Figure 1: Schematic of electron gun (50)

On an industrial scale, accelerators are used to generate electrons. When the generated electron-beam is directed at a target consisting of a high-atomic-number metal, such as tungsten or gold, X-rays with a broad spectrum of energies will be produced. The amount of energy absorbed, also known as the dose, is measured in units of kiloGrays (kGy), where 1 kGy is equal to 1,000 Joules per kilogram, or MegaRads (MR or Mrad), where 1 MR is equal to 1,000,000 ergs per gram. The accelators are generally described in terms of their energy and power. The low-energy accelerators range from 150 keV to 2.0 MeV while the medium-energy accelerators have energies between 2.5 and 8.0 MeV. High-energy or electron linear accelerators have beam energies above 9.0 MeV. Accelerator power is a product of electron energy and beam current. Available beam powers range from 5 to about 300 kW, for example a 5.0 MeV accelerator at 30 mA will have a power of 150 kW.(34)

Ionizing radiation is radiation that can ionize a molecule. The term ionizing radiation usually refers to the type of radiation that will ionize oxygen in air. Therefore, it is, radiation with a wavelength shorter than 253 nm, which includes x-rays generated in EB-curing systems. Unlike UV energy, EB energy ionizes anything in its path until all electrons are absorbed.(50) In general, EB radiation of materials begins when electrons with typical energies in the keV and MeV range are absorbed in matter and produce secondary electrons as a result of the energy degradation process. The fast electrons results in the formation of radicals, ions, trapped electrons and excited states of molecules or atoms through a Coulomb interaction of these secondary electrons with the atoms or molecules of the absorber. As a result, these fast electrons are able to initiate chemical changes in materials and modify them. Details on the principles and chemistry of ionizing radiation are well described elsewhere. (10,26,50)

For more than 40 years, such kinds of modifications have been used to change polymer structures and properties.(32) Structure and property modifications such as grafting, crosslinking and degradation of polymers can be induced by these electrons and by other ionizing radiation. Modification of the surface properties of polymers by grafting is achieved by graft copolymerization with different monomers. Grafting of polymers can be accomplished by electron beam irradiation of the polymer backbone to form radical sites, which then react with monomers present as liquid or vapor. Cross-linking usually tends to improve mechanical and thermal properties and chemical, environmental and radiation stabilities of materials. It occurs when two radicals produced on neighboring polymer units recombine. The relative molecular mass of the macromolecule increases and the melting point rises. Simultaneously to cross-linking, polymer degradation may take place by chain scission, which can lead to a decrease of the molecular mass. However, in some materials only one of these effects may be predominant during the radiation process.(16) Various applications of EB in achieving desired modifications mentioned above are presented elsewhere.(14,16,18,21,22,25,33,47)

1.2 Electron beam curing

EB curing is defined as "the use of electron beams as an energy source to induce a rapid conversion of especially formulated 100% reactive liquids to solids".(33) Free radical or cationic polymerization is initiated by the fast electrons and then followed by intensive crosslinking. In such way, dense polymer networks with tremendous abrasion, scratch and chemical resistance are produced.

Cationic polymerization differs from classic radical polymerization in several ways. The polymerization is not affected by oxygen, therefore curing can be done in air without a nitrogen blanket. The initiating species can be a stable chemical compound (including a proton or a carbonium ion) and usually can endure much longer than a free radicals. A considerable post-cure effect is observed; cationic polymerization results in a slower curing process than with free radicals. The initiating species can migrate over macroscopic distances even in opaque materials and in the dark (i.e. after radiant energy exposure stops). Cationic polymerization is also often supported by thermal activation.

Besides all the applications mentioned above, the most important and growing application of fast electrons with typical energies between 120 and 300keV (electron beam) is curing of solvent-free monomer/oligomer coatings, paints and printing inks.(33) Although EB curing technology is a long established, 30-year-old technology with solid advantages, the curing units have traditionally been cost prohibitive. Dump truck sized curing units were criticized for consuming; additionally, these units commonly cost around seven figures, a significant capital investment. Modification and calibration of the curing unit to collaborate with an existing process line also has been a huge contribution in this prohibitive cost. (37)

The recent increased interest in this application is, in part, a result of the recent availability of lower-voltage, lower-cost EB units.(36) For flexible packaging material, e-beam curing offers a number of advantages, especially to the converters. A high speed curing process that instantly dries up all coatings or ink in-line, producing 100% solid coatings, results in very high throughput rates. The lack of photoinitiator for curing the electron beam-based coatings/inks results in lower-cost formulation than UV-based coatings or inks. The utilization of energy in EB curing is also reported to be more efficient than the forced-air drying tunnels for water-based or and solvent-based cured coatings.(27) Unlike thermal curing, strict temperature or moisture controls may not be needed as only a moderate increase of temperature (about 15°C) occurs during the curing.

Other than that, EB curing offers competitive treatment cost per unit of product compared to more conventional chemical processes and it also produces hard, high gloss, stain, chemical and abrasion resistant finishes, especially for coatings.(21,22) The use of volatile or toxic chemicals can be avoided since uses solventless inks and varnishes. This results in very low to no odor and extractables, and complies with the environmental and FDA regulation. In addition, there is no need for oxidizers, incinerators or solvent-waste disposal as required for solvent-based systems.(27,35) Less space than for drying ovens associated with water- and solvent-based systems is now required for a compact e-beam curing unit, which can easily be fit in any desired place in the production or printing line (Figure 2 & 3).(28,45)

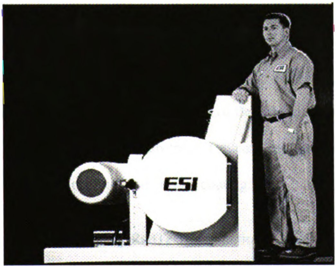


Figure 2: EZCure electron EB curing unit by Energy Science, Inc.(28)

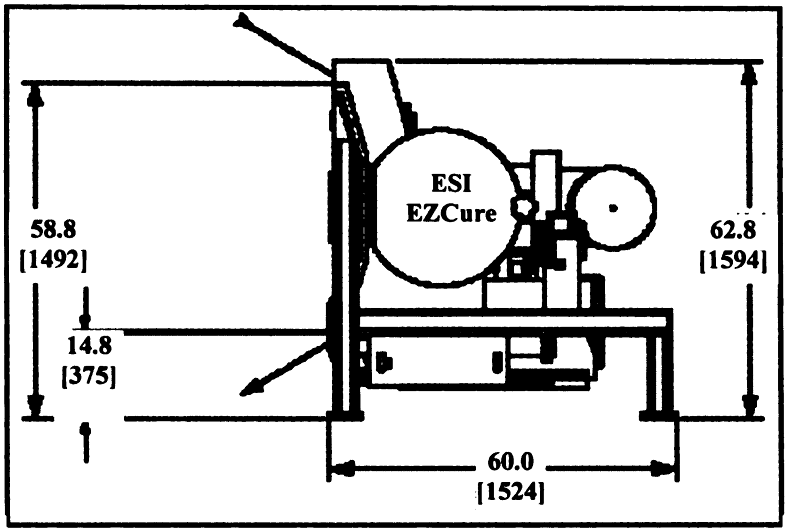


Figure 3: EZCure electron EB curing unit dimensions in inch [mm]. (28)

1.3 EB- curable coatings

EB-curable coatings generally consist of solventless liquids that can be applied to a substrate and converted into a solid, adherent film within a fraction of a second upon exposure to a beam of electrons. The coatings are combinations of oligomers (polymers with low molecular weights) and monomers, which control the viscosity before curing. Typical oligomers are acrylated urethane polyesters, acrylated epoxies and polyesters. A typical multifunctional monomer is trimethylolpropane triacrylate (TMPTA).(16) Flat surfaces such as paper sheets or webs, plastic sheets or flexible films, plastics, metal and wood are common materials used as the substrate. Typical application techniques for EB-cured coatings are spray, roll coating, and curtain coating.(44)

According to a survey done by RadTech International, one of the most significant applications of EB-curable coatings is in graphic arts coatings.(13,17) Accounting for about 25% of the use of all UV/EB formulated products, this application is the single biggest end user, with over 19,000 metric tons of product by year 2001. Many survey respondents indicated that EB overprint coatings applications including flexible film, foil and board offer good potential growth and graphic arts applications (including coatings and inks) for food packaging represents a big potential growth opportunity.

Issues of health and safety concerns in using electron beam curable coatings for food packaging applications were explained by Dr. Don Duncan, Director of Research at Wikoff Color Corporation. According to him, the EB coatings, when properly applied and cured, are fully suitable for most food packaging applications where three conditions apply: first, there is no intent of direct contact; second, the UV/EB print is separated from the food by a "functional barrier"; and third, the use of the UV/EB inks or coatings results in a food package free of odor or taint.(20)

1.4 Electron Beam curable coatings versus film lamination

Flexible film used as packaging materials in confectionary packaging for instance, involves the use of reverse-side-printed oriented polypropylene (OPP), which is then adhesive-laminated to another opaque film. A cold-seal adhesive is often then applied to the backside of the opaque film for subsequent cold sealing after filling. Replacing this bi-layer laminate structure with a single ply film (e.g. a direct-printed mono-web of thicker OPP film) and an EB 'top coat' delivers significant savings in material cost to the converter and the end-user alike. EB is used to cure these overprint coatings with properties strong enough to replace a layer of that opaque film. Additionally, since the film will be wound on itself with the cold-seal adhesive on its backside, the EB-curable coatings must also have excellent block resistance. (27)

Another practical objective in using EB-curable coatings is to replace an outer film in constructions in which the outer film functions only for graphics protection.(18,27) The EB-curable coatings, when applied over the printing, result in improved scratch and scuff resistance and provide high gloss, are odor free because they contain no photoinitiator, and provide a manageable coefficient of friction (CoF). In addition to the material savings realized from replacing a layer of film, there are the cost reductions realized from streamlining the printing and converting process. Many laminated structures take days to complete while EB cured coatings are processed instantly in-line, thus allowing the converter to print and ship the product in the same day.(27,28)

A cost estimation on the use of EB coating versus lamination was done by Rick Sanders of Energy Sciences, Inc. to provide information to the converter and end-user on how EB coating can yield significant savings in material cost (Table 1).(45)

Table 1: Comparison of material cost estimation between EB coatings and bi-layer film lamination

EB COATINGS vs. LAMINATIONS		
Replace Two-Ply Laminations with Monoweb and EB Overprint Varnish		
Assumptions:		
Product Width	36 inch Wide	
Production Hours	4,000 hours per year	
Line Speed	500 feet per minute	
Total Annual Production	51.8 MSI	
	Current Structure	**Proposed Structure**
	50G BOPP/RP/ADH/1.4 Mils BOPP	2.0 mil BOPP/Surface Print/EB OPV
	$ per 1,000 Square Inches (MSI)	$ per 1,000 Square Inches (MSI)
Cost of 50G & 1.4 Mils OPP	$0.1207	-----
Solvent-Based Adhesive	$0.0100	-----
Cost of 2.0 Mil OPP	-----	$0.1000
EB OPV @ $4.00/lb And 1.8 lbs/ream	-----	$0.0160
Total	$0.1307 per MSI	$0.1160 per MSI
Total Cost per Year	$6,770,260	$6,008,800
Net Savings per Year		$761,460

Source: Ink World Magazine, February 2003

The economic and toxic reduction assessment for Metallized Products, Inc. (MPI) in Manchester proved that the use of EB-coatings for their product resulted in a huge economic advantage when compared to thermal coating.(49) Even though a large capital investment was required for purchasing and installing the EB-unit, MPI was able to recover their investment, on their second EB-unit, over the course of two years because numerous factors affected annual operating costs, such as production speed and energy cost.

Even though converting from conventional coating systems to electron beam cured coating systems has proven to offer numerous advantages, development of this technology is still in the early stages compared to other EB curing applications. Only a small amount of literature related to EB-cured coatings on polymer substrates is available. Recent articles and papers on EB curing of coatings mostly emphasized advantages of converting from the use of conventional coatings to the EB-curable coatings.

The available studies from late 80s throughout the 90s on EB curing or processing of newly-developed polymers mostly provide ideas on correlation of some parameters such as degree of curing and radiation doses to the mechanical and chemical properties of the electron beam-cured samples.

1.5 Effect of electron beam curing on properties of polymer films

As EB curing is expected to result in modifications of at least the polymer surface, a primary objective of most of the studies was to examine how far EB curing will affect the properties and functionality of the cured polymers. Selected mechanical properties such as tensile properties, hardness, and film structure plus migrational behavior (total/overall migration) of the EB-cured samples were thoroughly discussed.

In a study on the effects of ionizing treatments (gamma photons & electron beam) on food simulant-packaging material combinations, Pillette(41) proved that the electron beam treatment (energy of 6 MeV) did not show any statistically significant difference in the structural, physicochemical and mechanical properties of the combinations, control and treated films. No statistically significant differences were also found for combinations in the total migration test in alcohol:water simulants. Very small total migration was also found within the limit of the detection level (<1mg/dm^2). The chemical analysis of volatiles showed the formation of hydrogen and methane. For that reason, some "microscopic" modifications were expected in the films due to the treatment.

The effect of EB radiation dosage and sensitizer (tri-methylolpropane trimethacrylate, TMPTMA) level on tensile properties of EVA was investigated by Datta.(19) The study showed that a higher amount of TMPTMA and higher dosage of radiation both had an adverse effect on the mechanical properties of EVA. Tensile strength and elongation at break depend on the degree of strain-induced crystallization which, in turn, depends on the polymer chain length and degree of crosslinking. Therefore, when crosslinking increases, the tensile strength may increase up to a certain level, beyond this level, the tensile strength is expected to decrease. A decrease in tensile strength might also result from a decrease in chain length due to chain scission. On the other hand, the modulus, which strongly depends on crosslinking density, was found to increase with increased degree of crosslinking.

Oliver et al (39) studied the mechanical properties of electron beam cured tripropylene glycol diacrylate (TPGDA) and propoxylated glycerol triacrylate (GPTA) films. The films were cured with two radiation doses (7.5 kGy and 105 kGy) and their tensile properties, including Young's modulus, were determined. For higher dose samples (105 kGy) , tensile stress rise was steep at low draw ratios, indicating a larger Young's modulus compared to the lower dose samples. As the electron beam curing dose increased, mechanical rigidity of the polymer network substantially increased, which is attributed to an increase of crosslinking density.

The acrylic resins used in the electron beam curing applications allow the widest latitude of formulation, besides representing the fastest curing systems. With proper formulation, acrylics can cure with doses as low as 1-5 Mrad. Even though the dose-to-cure is equally important for a variety of acrylic systems, this system is not highly dose-rate (current in mA) dependent. A study by Schroeter(46) on the effect of dose on properties for silicon resin mixtures consisting of various mixtures of acrylate and methacrylate monomer/polymer per thousand grams of silicon resin showed that the MEK-rub resistance obtained at any given dose is independent of dose rate. He also proved that properties such as solvent resistance and hardness of EB-cured resins improved as the total dose increases.

A similar study that supports Schroeter was done by Batten et al.(11) on electron beam curing of silicon-containing acrylates as a new surface-coating material. Performance and film-forming properties of a series of synthesized silicon-containing mono-, di-, tri- and tetraacrylates at increased curing doses were reported based on the results of similar physical tests; solvent rub resistance, pencil hardness and brittleness. From the results, they found that all tested silicon-containing acrylates cured rapidly at low doses. With increasing curing dose (2.5, 5, 10, 20, 40 and 60kGy), only tetraacrylates and triacrylates cured at low doses to give hard film with a high degree of flexibility while possessing good solvent resistance. On the other hand, monoacrylates polymerized to give highly viscous fluid, whereas diacrylates cured rapidly to give soft films.

Lox and Walden(30) did a study using a UV spectrophotometer to assess the effect of electron beam radiation upon migrational behavior of plastics. A single-sided migration test for 10 days at 40°C with ethanol:water was done on shrinkable PVC films radiated with increasing radiation doses from 3, 5, 7.5, 10, 20 and 25 kGy. A detailed explanation of how to measure migration and migrational behavior can be found in their previous study(31) and in a study by Lox et al.(29) The final result for total migration clearly demonstrated a drastic rise of migration rates at low-dose rate, probably due to the formation of small molecules. Conversely, at higher doses (>10 kGy), the migration rate decreases. The radiation process interferes with the pure migrational process (migration of un-radiated films) as less diffusible molecules were present after the high-dose exposure of the film.

A study on effects of electron beam treatment of the principal flexible food packaging materials (LDPE and OPP) by Gante and Pascatt(23) focused on chemical structure and mass transfer phenomena with an increasing dose of radiation. No significant changes were observed in the structure of polymer matrices or in oxygen permeability after films were treated at approved dose levels (<110kGy). However, several structural changes were observed by FTIR analysis at 100kGy and higher. With regard to total migration in ethanol:water simulants, all total migration data at increased doses were < 1 mg/dm^2, which is within the accepted limits of European Communities (EC) Regulations for polyolefins. The chemical analysis of volatiles from these films showed the presence of many different compounds in the treated film, which is comparable to the results from Lox and Walden.

As mentioned before, the EB-curable coating must have an excellent block resistance. Block resistance is the tendency of a coating not to adhere to another surface. An example of poor block resistance is when a part is painted and wrapped with paper, and the paper sticks to the "dry paint". If the paint is fully dry and not able to flow, the block resistance is usually good. Kauffman(27) proved that a commercially available EB coating for OPP for confectionary applications, termed as a release lacquer, exhibits low odor, high gloss, low COF and exceptional block resistance to a widely used cold-seal adhesive.

CHAPTER 2

MATERIALS AND METHODS

2.1 MATERIALS

All film samples were provided by Kraft. The tested substrates were 2 coextruded films (Substrates A and B) and 2 metallized polypropylene films (Substrates C and D). One overprint varnish, coded as C1, had been applied to the coextruded polypropylene, and a different varnish, C2, to the metallized films, and the varnishes were e-beam cured before receipt. Identical base films were also provided. The coated films were also printed with blue, white and blue plus white, each color in 50mm stripes at the center area of the films. The base films (uncoated and unprinted) were tested as controls, for comparison with the e-beam cured coated films.

Substrate A was a five-layer ethylene vinyl alcohol (EVOH) co-extruded film with a plastomer sealant consisting of co-polymer polypropylene (CoPP) / tie / EVOH / tie / linear low density polyethylene (LLDPE)-Metallocene sealant. Substrate B was a five-layer EVOH film coextruded with ethylene vinyl acetate (EVA) and cyclic olefin copolymer-LLDPE (COC-LLDPE) consisting of COC-LLDPE / tie / EVOH / tie / EVA. Both substrates had a gauge of 3.0 mils and were coated with C1 coating.

Both substrates C and D were metallized OPP with C2 coating. The differences between the two substrates were that substrate C was a vacuum-metallized, high barrier OPP film with proprietary sealant with 0.7 mil gauge, while Substrate D was an asymmetrical opaque barrier PP film metallized on one side and heat sealable on the other side, with 1.0 mil gauge.

2.2 METHODOLOGY

The mechanical properties tested included tensile properties, Elmendorf tear strength, dart drop impact strength, coefficient of friction, scuff resistance and heat seal behavior. The tests were performed according to standard ASTM methods, with appropriate modification.

The chemical properties testing involved 95% ethanol and 10:90 ethanol/water, the FDA-recommended simulants for fatty foods and for aqueous, acidic and low-alcohol-containing foods, respectively, for migration testing. The extracted liquid was tested by spectrometry techniques, and the pattern of peaks from the cured coated materials compared to that from the base films.

2.2.1 MECHANICAL PROPERTIES TESTS

A) Tensile Properties

The test for tensile properties of the substrates was performed according to standard ASTM method D882-01 "Standard Test Method for Tensile Properties of Thin Plastic Sheeting."(1) This test method covers determination of tensile properties of plastics in the form of thin sheeting, including film less than 1.0 mm or 0.04 inch in thickness. In this test, tensile properties of the control films were compared to the treated films. For the treated films, there were three treated areas of interest in the film structure where the tensile strength was measured: coated, uncoated and printed. Printed samples were taken from the printed center area of the films. Uncoated samples were taken from the film edges. Coated samples were taken from the coated but unprinted area between the uncoated edge and the printed center.

Since the substrates were anisotropic materials, a total of ten specimens were tested from each substrate, five replicates each for the machine and cross directions. The thickness of each substrate (control, uncoated, coated and printed) was measured with an auto micrometer by Testing Machines, Inc. (model 549M) according to ASTM D 6988-03 "Standard Guide for Determination of Thickness of Plastic Film Specimens."(8) Based on ASTM D 6287-98 "Standard Practice for Cutting Film and Sheeting Test Specimens," (7) the JDC Precision Sample Cutter (Model 1-10) was used to cut the films into strips of uniform 25 mm (1.0 in) width and length at least 50 mm (2.0 in.) longer than the grip separation. The INSTRON 4201 machine with initial grip separation of 2 inch and crosshead speed of 20 in/min was used to obtain the specimens' break/peak load and elongation. Detailed procedures and apparatus used are described in the ASTM standard.

B) Coefficient of Friction (CoF)

The static and kinetic coefficients of friction (CoF) of the plastic films were determined according to the standard ASTM method D1894 "Static and Kinetic Coefficients of Friction of Plastic Film and Sheeting."(3) The method covers determination of coefficients of starting and sliding friction of plastic film and sheeting when sliding over itself or other substances at specified test conditions.

The Monitor/Slip Friction machine (model 32-06) by Testing Machines Inc. which is equipped with a moving sled with a stationary plane similar to Method A of assembly of apparatus in the ASTM standard was used in this test. This equipment has the ability to calculate and display both static and average kinetic COF during one test cycle. A square metal block sled of 200 grams weight, measuring 2.5 inch by 2.5 inch was used for testing both the control and the treated films. For the treated films, both unprinted and printed areas were randomly tested. Five specimens of 3 inch by 18 inch from each substrate were tested for both static and kinetic coefficient of friction. As the films may exhibit different frictional properties in their respective principal directions due to anisotropy or extrusion effects, the common practice of testing the specimens was employed, that is by testing with the film's machine direction on machine direction (MD on MD).

C) Tear Strength

The substrates' tear strength was measured using an Elmendorf tear tester using a similar method as for paper. The test for tear strength was performed according to the standard ASTM method D1922-03a "Propagation Tear Resistance of Plastic Film and Thin Sheeting by Pendulum Method" to determine the average force to propagate tearing through a specified length of plastic film or nonrigid sheeting after the tear has been started, using an Elmendorf-type tearing tester. Rectangular specimens were cut using an Elmendorf standard cutter.(4) The tearing force of both the EB-cured films and control films was measured in the machine direction (MD) and cross direction (CD). For the treated films, there were two treated areas of interest in the films' structure where the tearing force was measured: unprinted and printed. Printed samples were taken from the printed center area of the films while the unprinted samples were taken randomly from the unprinted area of the films.

The Elmendorf tearing tester (Model 60-100) by Thwing-Albert Instrument Company was used in this test. Determination of number of plies to be used in testing for each substrate differs based on several factors such as the tearing behavior and the average scale reading obtained from the test. Based on the standard, the maximum accuracy of the apparatus lies in the scale range from 20 to 60. Therefore, enough sandwiched specimens should be tested to produce a scale reading between 20 and 60.

The number of plies for Substrate A was based on the scale reading produced by its CD specimens since the MD did not produce a scale reading within the required range (20 – 60). A single ply was used for Substrate B based on its tear behavior (oblique tear in opposite direction). For both metallized films (Substrates C and D), the appropriate numbers of plies were selected to produce scale readings of 10 or 20 only, since both substrates are relatively weak. The maximum number of plies that fit into the instrument's clamp was not sufficient to produce the desired scale readings (20 -60).

D) Heat- Seal Strength

The heat-seal strength of both cured and control films was measured in the MD and CD according to the ASTM F88-00 "Standard Test Method for Seal Strength of Flexible Barrier Materials."(9) For the treated films, only printed and unprinted areas were evaluated. The unprinted specimens were randomly cut from the unprinted coated or unprinted uncoated areas on the films. The films were cut into 1-inch wide strips using a JDC Precision Sample Cutter (model JDC 1-10) and fin-sealed with seal width of 1.0 inch, under specified conditions of temperature, pressure and dwell time. Both sealers used are from SENCORP Systems, Inc., the hot jaw heat sealer (Model 12ASL) and the thermal impulse sealer (Model 16TP).

Samples of all substrates were sealed initially with sealing conditions provided by the manufacturers. However, due to different sealers used for sealing, the final sealing conditions were based on the strongest seal that could be achieved. The sealing conditions for all substrates are summarized in Table 2 below:

Table 2: Sealing conditions for all substrates

FILMS	SEALER TYPE	SEALING CONDITIONS
Substrate A	Hot Jaw Heat Sealer	Temperature: 240F Dwell time : 0.5 second Pressure : 25 psi
Substrate B	Hot Jaw Heat Sealer	Temperature: 250F Dwell time : 1.4 second Pressure : 25 psi
Substrate C	Hot Jaw Heat Sealer	Temperature: 230F Dwell time : 1.0 second Pressure : 25 psi
Substrate D	Thermal Impulse Sealer	Dwell time : 0.6 second Pressure : 30 psi

Seven sealed replicates were prepared for each substrate film: control, unprinted and printed. The INSTRON 5565 machine with grip separation rate of 12 inch per minute and initial grip separation distance of 1 inch was used to obtain the average and peak load from the seal profile (plot of force versus grip separation). The method used to hold the film specimens during the test was the unsupported tail method.

E) Scuff resistance

The Sutherland dry rub test was performed according to ASTM D 5264-98 "Standard Practice for Abrasion Resistance of Printed Materials by the Sutherland Rub Tester"(6) to determine the durability and abrasion resistance of only the printed surface of all substrates. Test films with dimensions of 2 x 7 inches were cut from the printed area of the substrate and attached to the device. Similar film samples were attached to a 4 lb test block used to rub the test specimen. This standard 4 lb test block produced a contact pressure of 1 lb per square inch. Then, both test films were rubbed against one another at controlled speed and controlled cycles in increment of 5 strokes up to 25 strokes. Once the rubbing stopped, the test film was examined to see any type of failure such as ink transfer, wearing or scratching of the printed surface.

Rubbing the same films against one another did not result in any difference in rub resistance, even with 25 strokes. Therefore, in order to allow much more in-depth abrasion testing, a material with a more abrasive surface was used and attached to the test block instead of the film samples.

A GA-CAT (Comprehensive Abrasion Tester (CAT) by Gavarti Associates Ltd. of Milwaukee, WI.) standard receptor A-4 was used and attached to the test block. The test film and the standard receptor were rubbed against one another under the same test conditions and the test film was observed for the type of failure. With the standard receptor, adequate differences in rub resistance of the printed films were established and comparison between the printed films with similar coatings was made.

F) Dart Drop Impact Test

This test was performed according to standard ASTM method D1709-01 "Impact Resistance of Plastic Film by the Free-Falling Dart Method."(2) The test determines the energy that causes different plastic films to fail under specified conditions of impact of a free-falling dart. This energy is expressed in terms of the weight (mass) of the missile falling from a specified height which would result in 50% failure of specimens tested.

The Test Method A with standard staircase testing technique was used in this study. Test Method A employs a dart with a 38 mm (1.5 in) diameter hemispherical head dropped from a height of 0.66 m (26 in). In the staircase testing technique, a uniform missile weight is employed during the test and the missile weight is decreased or increased by the uniform increment after the test of each specimen, depending upon the result (fail or not fail) observed for the specimen. The control films and the printed area of the treated films were cut into 6-inch square films with at least 20 replicates and tested using a dart missile weighing 48 grams and 15 gram rings as increments. The apparatus, testing procedure and calculation used are described in the ASTM standard.

2.2.2 CHEMICAL PROPERTIES

A) Migration testing

The polyolefin migration test involved the FDA-recommended simulants for fatty foods (95/100% ethanol) and for aqueous, acidic and low-alcohol-containing food (10:90 ethanol/water). The test was performed according to the USFDA Guidance for Industry of Chemistry Recommendations for Preparation of Food Contact Notifications and Food

Additive Petitions for Food Contact Substances (FCS)(51) and ASTM D 4754 "Standard Test Method for Two-Sided Liquid Extraction of Plastic Materials Using FDA Migration Cell."(5) To determine the total migration, substrates were stored at 40°C for 10 days in contact with the food simulants as recommended by the FDA. For each substrate, a single sample was prepared initially to examine the result using sampling periods of 24, 48, 120 and 240 hours. Based on the results of that single sample, triplicate samples were then prepared for the actual data. The amount of FCS that migrated from the samples was determined by weighing the samples before and after exposure to the simulants.

The remaining simulants were then tested using the Perkin Elmer (Lambda25) UV/VIS Spectrometer and Fourier Transform Infrared Spectroscopy (FT-IR) Spectrometer; model Spectrum 1000 also by Perkin Elmer. The absorbance of the remaining simulants was scanned from the whole region of the visible light wave length to the UV light wavelength (190 nm – 1100nm) to detect any appearance of peaks of the migrants in the simulants. In the FT-IR testing, the simulants were scanned from infrared light frequency of 515 cm^{-1} to 4000 cm^{-1} to observe the percent transmittance of the migrants. For both UV/VIS and FTIR, the pattern of peaks from the cured coated (treated) materials was compared to that from the base (control) films to possibly see indication of any new migrants from the cured coated film.

CHAPTER 3

RESULTS AND DISCUSSION

3.1 TENSILE PROPERTIES

In order to compare and verify any differences in tensile properties between the EB-cured films and the control films, three-way analysis of variance (ANOVA) was employed to analyze the results. The experiment has a three-way factorial design (4 x 4 x 2) with 5 replicates. Residual diagnostics was done to check the normality and distributional assumptions of this dataset.(48) The residuals plot showed no pattern indicating any violation of normal distributional assumptions that are required for statistical inference in this model.

The result from the ANOVA table with the lack-of-fit test in Type III fully adjusted sums of squares table shows that the 3-way interaction among all factors is significant (P-value of <0.0001) . That means the tensile strength measurement does depend on these three important factors. Therefore, inferences involving any particular factor were done within the combination of levels of the other two factors using the F-test with Type I error rate of 5% (see Appendix B).

Since the point of interest is to verify any differences in tensile strength between treatment levels, the summary of tensile strength mean differences between levels of treatment for each substrate and direction combination is presented in Table 3. All values were obtained at break except for substrate A in the CD, where the maximum value was obtained at yield.

Table 3: Tensile strength mean comparison between treatments, separately for each substrate in MD and CD.

SUBSTRATE	TREATMENT	Tensile Strength (lb/inch^{2})	
		MD	CD
SUBSTRATE A	Control	17,223^{a}	3,665^{a}
	Coated	13,768^{b}	3,043^{a}
	Uncoated	15,872^{a}	3,246^{a}
	Printed	13,369^{b}	3,069^{a}
SUBSTRATE B	Control	3,107^{a}	3,054^{a}
	Coated	2,873^{a}	2,965^{a}
	Uncoated	3,028^{a}	2,852^{a}
	Printed	2,814^{a}	2,836^{a}
SUBSTRATE C	Control	13,245^{a}	35,011^{a}
	Coated	16,377^{b}	34,111^{a}
	Uncoated	16,081^{b}	38,470^{b}
	Printed	14,167^{a}	30,177^{c}
SUBSTRATE D	Control	18,290^{a}	48,046^{a}
	Coated	16,149^{b}	42,016^{b}
	Uncoated	16,396^{b}	42,710^{bc}
	Printed	16,510^{b}	43,727^{c}

For each substrate and direction, different letters after TS indicate a significant difference ($P<0.05$) between treatments.

In general, significant differences in tensile strength as a function of treatment were found for substrate A, C and D films, but not for substrate B.

3.1.1 Load at Peak:

Since coating and printing slightly increases the thickness of the film, it will slightly decrease the calculated tensile strength if the coating or printing does not contribute any strength and the strength of the base film is unchanged. Therefore, as an alternative to analyzing tensile strength, the analysis can be done on the peak load. The same statistical method, three-way analysis of variance (ANOVA) and Type 1 error rate of 5%, was used. The summary of peak load mean differences between levels of treatment for each substrate and direction combination is presented in Table 4.

Table 4: Peak load mean comparison between treatments separately for each substrate in MD and CD.

SUBSTRATE	SAMPLE	Ave. Thickness (mil)	Peak Load MD (lbs)	Peak Load CD (lbs)
			Average	Average
SUBSTRATE A	Control	2.4	41.34^{a}	8.80^{a}
	Coated	3.0	41.30^{a}	9.13^{a}
	Uncoated	2.9	46.03^{b}	9.42^{a}
	Printed	3.1	41.44^{a}	9.51^{a}
SUBSTRATE B	Control	3.0	9.32^{a}	9.16^{a}
	Coated	3.0	8.62^{a}	8.90^{a}
	Uncoated	3.0	9.09^{a}	8.56^{a}
	Printed	3.1	8.72^{a}	8.79^{a}
SUBSTRATE C	Control	0.7	9.27^{a}	24.51^{a}
	Coated	0.7	11.46^{b}	23.88^{a}
	Uncoated	0.6	9.65^{a}	23.08^{a}
	Printed	0.8	11.33^{b}	24.14^{a}
SUBSTRATE D	Control	1.0	18.29^{a}	48.05^{a}
	Coated	1.1	17.76^{ab}	46.21^{c}
	Uncoated	1.0	16.40^{b}	42.71^{b}
	Printed	1.1	18.16^{a}	48.1^{a}

For each substrate and direction, different letters after Peak Load indicate a significant difference ($P<0.05$) between treatments.

The statistical analysis shows that there are significant differences in peak load between MD and CD of all substrates except substrate B. While some statistically significant differences were found between the control and one or more of the subcategories of treated films in all substrates except substrate B (see Appendix B), in the base comparison of whether there was a difference between the printed film and the control, a significant difference was found only for substrate C, as its peak load actually increased by more than 20%. Therefore, much of the difference found in the tensile strength comparison does, in fact, seem to be due to the increase in thickness attributable to the coating, rather than to any effect of the e-beam curing.

3.1.2 Elongation at break

Percent elongation at break for all substrates was also measured by dividing the change of the specimen's length by it's the initial jaw separation, which was 2.0 inches. The data were transformed to natural log in order to satisfy the normality and distributional assumptions required for statistical inference using this data. The same statistical method (three-way ANOVA) using Type 1 error rate of 5% confirmed that percent elongation at break between MD and CD for all substrates was significantly different except for substrate B. No significant difference of percent elongation between control and treated films was found for substrates B and D. The summary of percent elongation at break mean differences between levels of treatment for each substrate and direction combination is presented in Table 5.

Table 5: Mean comparison of percent elongation at break between treatments, separately for each substrate in MD and CD.

SUBSTRATE	TREATMENT	% Elongation at Break	
		MD	CD
SUBSTRATE A	Control	68.4^{a}	594^{a}
	Coated	86^{a}	$332*^{c}$
	Uncoated	96^{a}	$338*^{c}$
	Printed	93.4^{a}	$92*^{b}$
SUBSTRATE B	Control	549^{a}	624^{a}
	Coated	486^{a}	590^{a}
	Uncoated	466^{a}	566^{a}
	Printed	477^{a}	552^{a}
SUBSTRATE C	Control	109^{a}	44^{a}
	Coated	208^{b}	61^{a}
	Uncoated	148^{ab}	54.6^{a}
	Printed	181^{b}	50^{a}
SUBSTRATE D	Control	217^{a}	56^{a}
	Coated	239^{a}	67^{a}
	Uncoated	222^{a}	60^{a}
	Printed	235^{a}	64^{a}

For each substrate and direction, different letters after % elongation indicate a significant difference ($P<0.05$) between treatments.

* There was considerable variation in elongation at break for these samples. For coated and uncoated, one sample of each broke prematurely and results were excluded from the calculation. For the printed samples, 3 of the 5 samples broke at very low elongations, and are included in the average. The main reason for this is because substrate A is a machine direction oriented film; therefore when the strips were stretched in the CD, premature breakage was likely, resulting in variation in the results.

3.2 COEFFICIENT OF FRICTION (CoF)

An unusual result was obtained from the control films of substrates B, C and D where their kinetic CoF was found higher than their static CoF. Repeated tests were performed on the substrates to verify these values, and resulted in consistent CoF values. Therefore, these unusual results appeared to be the true CoF values for these substrates. The differences in the static and kinetic coefficient for both control and treated films were statistically compared. Using the two population t-test with Type 1 error rate of 5%, we found that that only Substrate C showed significant difference between control and treated films in both its static and kinetic CoF. The static CoF of control and treated films of Substrates B and D were found not significantly different, but both substrates showed significant differences in their kinetic CoF. For Substrate A, only its static CoF showed significant differences between the control and treated films. Where significant differences were found, the treated films had lower coefficients of friction than the control films. The summary of CoF mean comparison of the films is presented in Table 6 below:

Table 6: Mean comparison of the Coefficient of Static and Kinetic Friction for all substrates

SUBSTRATES	FILM	STATIC CoF	KINETIC CoF
SUBSTRATE A	Control	0.1690^{a}	0.1158^{a}
	Treated	0.1120^{b}	0.1088^{a}
SUBSTRATE B	Control	0.1958^{a}	0.3268^{a}
	Treated	0.2110^{a}	0.1616^{b}
SUBSTRATE C	Control	0.2584^{a}	0.3652^{a}
	Treated	0.1278^{b}	0.1238^{b}
SUBSTRATE D	Control	0.2970^{a}	0.3950^{a}
	Treated	0.2092^{a}	0.1830^{b}

For each substrate and direction, different letters after CoF indicate a significant difference ($P<0.05$) between treatments.

3.3 TEAR STRENGTH

The tearing force of each substrate was determined using this equation:

$$\text{Tearing Force, grams} = \frac{16 \times \text{Average Scale Reading}}{\text{Number Of Plies}}$$

The same statistical technique used in the tensile properties was employed to analyze the tearing force data, except that the experimental design for this dataset was a three-way factorial design (4 x 3 x 2) with 10 readings. These data were also transformed to natural log in order to satisfy the normality and distributional assumptions required for statistical inference with this data. The test results for each substrate are summarized in Table 7 below:

Table 7: Tearing force mean comparison between treatments, separately for each substrate in MD and CD.

SUBSTRATE	FILM	TEARING FORCE (gf)	
		MD	CD
SUBSTRATE A (3 PLIES)	Control	53.07^{a}	127.73^{b}
	Unprinted	50.67^{ab}	117.33^{a}
	Printed	48.27^{b}	121.87^{a}
SUBSTRATE B (1 PLY)	Control	554.40^{b}	154.40^{a}
	Unprinted	743.30^{a}	152.80^{a}
	Printed	722.40^{a}	117.60^{b}
SUBSTRATE C (30 PLIES)	Control	6.91^{a}	4.05^{b}
	Unprinted	5.92^{b}	4.27^{a}
	Printed	5.65^{b}	4.24^{a}
SUBSTRATE D (30 PLIES)	Control	11.09^{a}	7.12^{b}
	Unprinted	9.04^{b}	5.55^{a}
	Printed	9.23^{b}	5.68^{a}

For each substrate and direction, different letters after Tearing Force indicate a significant difference ($P<0.05$) between treatments.

From the analysis, we found that all substrates showed significant differences in tear strength between the control and treated (printed & unprinted) films as well as between MD and CD. However, there was no pattern to the behavior. Tear strength increased with treatment for substrate B in the MD and C in the CD, while it decreased for substrates A and D, and for substrate B in the CD and substrate C in the MD.

3.4 HEAT-SEAL STRENGTH

The peak seal strength and average seal strength data are presented in Appendix A, Table A.6.1 and Table A.6.2. Since the peak strength data shows a lot more variation than the average strength, the peak strengths were statistically analyzed using three-way analysis of variance (ANOVA) with Type 1 error rate of 5%. In order to assure the normality and distributional assumptions required for statistical inference of this data, the peak strength data were transformed to natural log before further analysis. From the analysis, we found that only substrate B showed no difference in peak seal strength between its MD and CD films, while the other substrate's MD and CD peak seal strengths were significantly different from each other. Significant differences were found between control and printed films for substrates A, C, and D in the MD (see Appendix). For A, seal strength increased, while for C and D it decreased. All other differences were not significant. The summary of peak seal strength mean differences between treated and control films for each substrates with MD and CD combination is presented in Table 8.

Table 8: Peak seal strength force mean comparison between treatments, separately for each substrate in MD and CD.

SUBSTRATE	DIRECTIONS	PEAK SEAL STRENGTH (lbs/inch)		
		CONTROL	UNPRINTED	PRINTED
SUBSTRATE A	MD	13.8386^{a}	15.6271^{a}	23.4300^{b}
	CD	8.6171^{a}	10.6086^{a}	9.6671^{a}
SUBSTRATE B	MD	9.6314^{a}	9.7043^{a}	10.5700^{a}
	CD	8.9600^{a}	9.5914^{a}	9.1729^{a}
SUBSTRATE C	MD	0.8729^{a}	0.7629^{a}	0.5929^{b}
	CD	0.6543^{a}	0.5286^{a}	0.5257^{a}
SUBSTRATE D	MD	2.0657^{a}	1.6314^{ab}	1.4100^{b}
	CD	2.1357^{a}	2.2129^{a}	2.1114^{a}

For each substrate and direction, different letters after Peak Seal Strength indicate a significant difference ($P<0.05$) between treatments

The test strip failure modes were also identified in this test. Based on the ASTM standard, seven modes of failure were illustrated and categorized into failure and types. The modes were numbered (Table 9) to make it easier for reference.

Table 9: Modes of Failure

Number	Failure	Type
1	Seal	Adhesive (peel)
2	Material	Cohesive
3	Material	Delamination
4	Material	Break
5	Material	Break/Tear (remote)
6	Material	Elongation
7	Seal + Material	Peel + Elongation

Most of the MD films of control substrate A experienced Modes 1and 3, while the CD films experienced a combination of Modes 1, 2 and 6. All MD films of unprinted substrate A failed in Mode 1 while the printed films failed in both Modes 1 and 3. All treated CD films failed in Mode 4. For substrate B, all films, control, unprinted and printed, in both MD and CD failed in a combination of Modes 1 and 6. All substrate C control MD films failed in a combination of Modes 1 and 2, while most of its CD films failed in a combination of Modes 1 and 3. For most of treated substrate C, both MD and CD films failed in a combination of Modes 1 and 4. Substrate D control films, both MD and CD, experienced a combination of Modes 1, 3 and 4. As for its treated films (unprinted and printed), both MD and CD films mostly experienced failure in Modes 1 and 4.

3.5 SCUFF RESISTANCE

Comparison was made between two substrates with similar coatings which had undergone identical test conditions. This means that the abrasion resistance of printed Substrate A was compared to printed Substrate B and the abrasion resistance of printed Substrate C was compared to printed Substrate D. For every 5 stroke increment, adequate differences of rub resistance between the two different tested substrates were identified and compared.

According to the Packaging Institute(39), ink transfer may be defined as the presence of ink residue on a portion of the test strip other than where it was printed. Wearing may be defined as abrasion of a printed ink film. Scratching occurs where a relatively deep, sharply defined cut is made in the ink film. Based on these definitions, qualitative evaluations of each pair of substrates at 5 strokes and 25 strokes are presented in Tables 10 and 11.

Table 10: Qualitative Rub Resistance Comparison of Substrate A and Substrate B

Number of Strokes	Substrate A	Substrate B
5	Appearance of wearing and deep scratching on one third of film surface.	Appearance of wearing and only light scratching on film surface.
25	Extensive wearing and deep scratching on the overall surface.	Increased wearing and light scratching in 2/3 of the surface but no obvious deep scratching.

Table 11: Qualitative Rub Resistance Comparison of Substrate C and Substrate D

Number of Strokes	**Substrate C**	**Substrate D**
5	Appearance of extensive wearing and light scratching on overall film's surface.	Appearance of wearing and light scratching only on 2/3 of film's surface.
25	Increased extensive wearing and appearance of deep scratching on the overall surface.	Increased wearing and light scratching on 2/3 of the surface but no obvious deep scratching.

From the qualitative results above, it can be concluded that Substrate B has higher rub resistance than Substrate A and Substrate D has higher rub resistance than Substrate C.

3.6 DART DROP IMPACT

Complete data for each dart drop test for all substrates are listed in Table A.7.1 to Table A.7.4 in Appendix A. These tables illustrate failure and non failure results at each tested weight along with the calculated dart impact failure weight for all substrates. The impact failure weight for each substrate was determined using the following equation:

$$W_F = W_O + \left[\Delta W\left(\frac{A}{N} - \frac{1}{2}\right)\right]$$

where : W_F = impact failure weight, (g)

W_O = missile weight to which an *i* value of zero is assigned.

ΔW = missile weight increment (15 gram)

A = total of in_i's

N = total number of failures

A sample of a chart and dart impact failure weight determination of one of the films is presented in Appendix B. The summary of the dart drop impact test results is presented in Table 12 below:

Table 12: Summary of the Dart Drop Impact test results.

SUBSTRATE	FILM	N	A	Wo	ΔW	W_F (g)
SUBSTRATE A	Control	10	5	63	15	63
	Printed	10	9	63	15	69
SUBSTRATE B	Control	10	28	168	15	202.5
	Printed	10	22	153	15	178.5
SUBSTRATE C	Control	10	9	63	15	69
	Printed	10	5	63	15	63
SUBSTRATE D	Control	10	14	138	15	151.5
	Printed	10	9	153	15	159

From the results, the printed films of Substrate A and D showed slightly higher impact strength than the control films, whereas for Substrate B and C, the control films showed higher impact strength than the printed films. Nevertheless, impact strengths between the control and printed films of all substrates showed only very small differences from each other.

3.7 TOTAL MIGRATION

The amount of FCS that migrates from the substrates to the simulants was determined by weighing the samples, before and after exposure to the simulants. The total migration for the substrates was calculated and expressed in milligrams of migrant(s) per square decimeter of sample exposed, E using the equation from ASTM Standard D 4754 as below:

$$E = \frac{(W - B)}{\left[(2\pi R^2 + CT)N\right]}$$

where:
W = initial weight of plastic film disk (before exposure), mg.
B = final weight of plastics film disks (after exposure), mg.
R = Radius of the disk, m
C = Circumference of disk, m.
T = Thickness of disk, m.
N = Number of disks per cell

Complete results of the substrates weight difference and the total migration are presented in Table A.8.1 to A.8.8 in Appendix A. Table A.8.1 to A.8.4 shows the results for substrates A and B exposed to 10% and 95% ethanol:water simulant and Table A.8.5 to A.8.8 are the results for substrates C and D.

The results of both metallized films (Substrates C and D) clearly showed no pattern of behavior in their weight differences when exposed to both 10% and 95% ethanol. Some of the samples showed both decrement and increment of weight over time. For instance, the metallized coatings of the control films of Substrate C were observed to be flaking off when exposed to 10% ethanol for 120 and 240 hours, which probably explains the increase in weight difference for these samples. On the other hand, the metallized coating of all samples were still intact when exposed to 95% ethanol. While increased weight loss over time was found in treated C samples exposed to 10% ethanol, the reverse result was found for samples exposed to 95% ethanol.

Two of the control films of substrate D exposed to 10% ethanol: water showed no difference in weight while a consistent decrease in weight over time was found when exposed to 95% ethanol. The treated films of substrate D exposed to 10% ethanol showed high decrement in the 24 hours samples, followed by constant weight difference over time for 48 and 120 hour samples. However an unexpected weight gain was found in the 240 hour sample. When exposed to 95% ethanol, both control and treated samples showed inconsistent weight loss over time.

Due to the metallized coating flaking-off the substrates and the inconsistencies of the data, it is not possible to use the samples' weight difference as FCS amount to further calculate the total migration of the substrates. Results showed that migration was in both directions (from substrate to simulant and from simulant to substrate). For that reason, no further investigation of the total migration of Substrate C and D were done since this was beyond of the scope of this study. Hence, the rest of the discussion on migration behavior will only focus on total migration of the co-extruded films: substrate A and substrate B.

The results for all films from both substrates A and B showed a decrease in weight after being exposed to the both simulants. Thus, the films' weight differences, which depict the amount of FCS that migrates from the films, were used to calculate the total migration. The total migration data for both substrates were statistically analyzed using the three-way analysis of variance (ANOVA) with Type 1 error rate of 5%. To simplify the interpretation of the data, total migration in 10% and 95% ethanol:water simulant were analyzed separately. Only the data for total migration in 95% ethanol:water simulant were transformed to natural log in order to satisfy the normality and distributional assumption requirements. The main interest of this analysis is to verify any differences in total migration between the control and treated films of each substrate at each time of exposure (24, 48, 120 and 240 hours). The summary of total migration mean differences of each substrate in 10% and 95% ethanol:water simulants are presented in Table 13 and Table 14, respectively.

Table 13: Total migration mean comparison for substrates A and B in 10% ethanol:water simulant.

SUBSTRATES	**EXPOSURE (hours)**	**AVERAGE TOTAL MIGRATION (mg/dm^2)**	
		Control film	**Treated film**
SUBSTRATE A	24	0.9347_a	0.6301_a
	48	0.4440_b	0.5134_b
	120	0.3505_c	0.4434_c
	240	0.5842_d	0.6301_d
SUBSTRATE B	24	1.2835_e	1.4702_e
	48	0.9801_f	0.7468_f
	120	1.0735_g	0.9335_g
	240	1.4235_h	1.7269_h

For each substrate, different subscripted letters after Total Migration indicate a significant difference ($P<0.05$) between films within hours.

Table 14: Total migration mean comparison for substrates A and B in 95% ethanol:water simulant.

SUBSTRATES	**EXPOSURE (hours)**	**AVERAGE TOTAL MIGRATION (mg/dm^2)**	
		Control film	**Treated film**
SUBSTRATE A	24	1.5189_a	0.7234_b
	48	0.8880_c	0.9568_c
	120	0.9814_d	1.1201_d
	240	1.0749_e	1.2368_e
SUBSTRATE B	24	3.2204_f	2.3570_f
	48	1.7036_g	1.5402_g
	120	1.9836_h	1.4935_h
	240	2.3103_i	2.4503_i

For each substrate, different subscripted letters after the Total Migration indicate a significant difference ($P<0.05$) between films within hours.

From the analysis, no significant difference was found between the control and treated films of both substrates A and B exposed in 10% ethanol: water simulant, within each time of exposure. As for substrates exposed in 95% ethanol: water simulant, the control and treated films of both substrates also showed no significant difference from each other except for films from substrate A exposed for 24 hours.

The analysis also indicated some significant differences in total migration between time of exposure for control and treated films of each substrate. However, with the inconsistent pattern of the data, it is not possible to draw any concrete conclusions.

The UV/VIS and the FTIR spectrums of the remaining simulants from the migration test for control versus treated films of all substrates are illustrated in Appendix C and Appendix D, respectively. Figure C.1.1 to Figure C.4.8 are the UV/VIS spectrums while Figure D1.1 to Figure D.4.8 are the FTIR spectrums. When comparing the simulants from the substrates' control films with treated films, the patterns of both UV/VIS and FTIR spectrums noticeably illustrated differences between the films. This difference also means that there are indications of new migrants from the treated films compared to that control films. These indications can be verified by further identifying all the migrants; however this is beyond the scope of this study.

CHAPTER 4

CONCLUSION AND RECOMMENDATIONS

The difference in selected mechanical and chemical properties between the control and treated film of all substrates was demonstrated and discussed in detail in the previous chapter. Even though most of the tests showed significant differences in the mechanical properties between control and treated films, these differences are relatively small and will not result in major changes in performance. As for the chemical properties, the total migration of the control and treated films of both substrates A and B were found to not significantly differ from each other even though the pattern of the total migration data was found inconsistent over time. For that reason, it can be said that e-beam curing does not have much impact on the migrational behavior of these substrates. With these results, it can be concluded that the e-beam cured films should be suitable for many flexible packaging applications.

In the case of substrates C and D, the chemical properties could not be described by only total migration determination. A single-sided migration test where only the sealant or heat-sealable side of the substrates is exposed to the food simulants is highly recommended for substrate C and D to avoid the problem of the metallized coating flaking off the substrate during the migration period. In addition, specific migration tests would be useful to further investigate substances that migrate from the substrates to the food stimulant. An indication of new migrants was found in the treated films when comparing their UV/VIS and FTIR spectrums with the control films. Testing the remaining simulants through the GC-mass spectrometry is recommended in order identify peaks of significant size that differ between the control and the treated films.

APPENDIX A

TEST DATA AND RESULTS

Table A1: Tensile Strength Data

SUBSTRATES	DIRECTIONS	AVERAGE & STANDARD DEVIATION	TENSILE STRENGTH (psi)			
			Control	Coated	Uncoated	Printed
SUBSTRATE A	MD	Average	17,223	13,768	15,872	13,369
		Std Deviation	1,579	256	591	889
	CD	Average	3,666	3,043	3,247	3,069
		Std Deviation	178	32	39	66
SUBSTRATE B	MD	Average	3,108	2,874	3,028	2,814
		Std Deviation	102	56	157	66
	CD	Average	3,054	2,965	2,853	2,836
		Std Deviation	117	70	90	95
SUBSTRATE C	MD	Average	13,245	16,377	16,081	14,168
		Std Deviation	4,145	1,222	3,120	1,285
	CD	Average	35,011	34,111	38,470	30,178
		Std Deviation	3,149	833	1,429	1,712
SUBSTRATE D	MD	Average	18,290	16,149	16,396	16,511
		Std Deviation	1,287	725	891	452
	CD	Average	48,046	42,016	42,710	43,727
		Std Deviation	1,023	479	1,291	1,287

Table A2: Load at Peak Data

SUBSTRATES	DIRECTIONS	AVERAGE & STANDARD DEVIATION	LOAD AT PEAK (psi)			
			Control	Coated	Uncoated	Printed
SUBSTRATE A	MD	Average	41.336	41.304	46.03	41.444
		Std Deviation	3.7904	0.7671	1.7140	2.7547
	CD	Average	8.7974	9.1296	9.4162	9.5152
		Std Deviation	0.4271	0.0948	0.1123	0.2048
SUBSTRATE B	MD	Average	9.3228	8.6206	9.0854	8.7248
		Std Deviation	0.3051	0.1692	0.4700	0.2032
	CD	Average	9.162	8.8954	8.5582	8.7922
		Std Deviation	0.3505	0.2109	0.2707	0.2946
SUBSTRATE C	MD	Average	9.2716	11.464	9.6486	11.334
		Std Deviation	2.9016	0.8552	1.8722	1.0280
	CD	Average	24.508	23.878	23.082	24.142
		Std Deviation	2.2046	0.5829	0.8572	1.3699
SUBSTRATE D	MD	Average	18.29	17.764	16.396	18.162
		Std Deviation	1.2871	0.7971	0.8913	0.4973
	CD	Average	48.046	46.218	42.71	48.1
		Std Deviation	1.0235	0.5268	1.2908	1.4154

Table A.3: Percent Elongation at Break Data

SUBSTRATES	TREATMENT	% Elongation at Break	
		MD	CD
SUBSTRATE A	Control	68.4	594
	Coated	86	332
	Uncoated	96	338
	Printed	93.	92
SUBSTRATE B	Control	549	624
	Coated	486	590
	Uncoated	466	566
	Printed	477	552
SUBSTRATE C	Control	109	44
	Coated	208	61
	Uncoated	148	54.6
	Printed	181	50
SUBSTRATE D	Control	217	56
	Coated	239	67
	Uncoated	222	60
	Printed	235	64

Table A.4: Coefficient of Friction Data

SUBSTRATES	AVERAGE & STD.DEV.	STATIC COF		KINETIC COF	
		CONTROL	PRINTED	CONTROL	PRINTED
SUBSTRATE A	Average	0.169	0.112	0.1158	0.1088
	Std.Deviation	0.0509	0.0074	0.0696	0.0722
SUBSTRATE B	Average	0.1958	0.211	0.3268	0.1616
	Std.Deviation	0.0665	0.0718	0.0348	0.0929
SUBSTRATE C	Average	0.2584	0.1278	0.3652	0.1238
	Std.Deviation	0.0241	0.0233	0.0455	0.1007
SUBSTRATE D	Average	0.297	0.2092	0.395	0.183
	Std.Deviation	0.0635	0.1060	0.0180	0.1365

Table A.5: Elmendorf Tear Strength Data

SUBSTRATES	DIRECTIONS	AVERAGE & STD.DEV	TEARING FORCE (gf)		
			Control	Unprinted	Printed
SUBSTRATE A (3 PLIES)	MD	Average	53.07	50.67	48.27
		Std Deviation	7.69	11.86	7.59
	CD	Average	127.73	117.33	121.87
		Std Deviation	3.43	8.62	13.77
SUBSTRATE B (1 PLY)	MD	Average	554.40	743.36	722.40
		Std Deviation	22.33	29.23	37.72
	CD	Average	554.40	152.80	117.60
		Std Deviation	22.33	27.83	9.28
SUBSTRATE C (30 PLIES)	MD	Average	6.91	5.92	5.65
		Std Deviation	0.32	0.33	0.25
	CD	Average	4.05	4.27	4.24
		Std Deviation	0.30	0.44	0.27
SUBSTRATE D (30 PLIES)	MD	Average	11.09	9.04	9.23
		Std Deviation	0.36	0.53	0.31
	CD	Average	7.12	5.55	5.68
		Std Deviation	0.22	0.33	0.31

Table A.6.1: Peak Seal Strength Data

SUBSTRATES	DIRECTIONS	Average & Std Deviation	PEAK SEAL STRENGTH (lbs/inch)		
			CONTROL	UNPRINTED	PRINTED
SUBSTRATE A	MD	Average	13.8386	15.6271	23.4300
		Std Deviation	3.8860	5.3354	1.5041
	CD	Average	8.6171	10.6086	9.6671
		Std Deviation	1.9215	1.3752	2.3547
SUBSTRATE B	MD	Average	9.6314	9.7043	10.5700
		Std Deviation	0.2061	0.2178	0.2605
	CD	Average	8.9600	9.5914	9.1729
		Std Deviation	0.3525	0.2785	0.3138
SUBSTRATE C	MD	Average	0.8729	0.7629	0.5929
		Std Deviation	0.0256	0.2179	0.1643
	CD	Average	0.6543	0.5286	0.5257
		Std Deviation	0.1373	0.0717	0.1091
SUBSTRATE D	MD	Average	2.0657	1.6314	1.4100
		Std Deviation	0.4605	0.3360	0.5996
	CD	Average	2.1357	2.2129	2.1114
		Std Deviation	0.7790	0.6515	0.4418

Table A.6.2: Average Seal Strength Data

SUBSTRATES	DIRECTIONS	Average & Std Deviation	AVERAGE SEAL STRENGTH (lbs/inch)		
			CONTROL	UNPRINTED	PRINTED
SUBSTRATE A	MD	Average	5.4143	6.2829	5.9486
		Std Deviation	2.2301	2.4390	5.9486
	CD	Average	5.1900	5.2857	6.2957
		Std Deviation	2.3925	2.7141	3.1553
SUBSTRATE B	MD	Average	8.9700	9.0571	9.4886
		Std Deviation	0.3158	0.1762	0.1833
	CD	Average	7.9971	8.1614	8.3186
		Std Deviation	0.1292	0.1767	0.1129
SUBSTRATE C	MD	Average	0.5000	0.5643	0.3600
		Std Deviation	0.1519	0.1433	0.1274
	CD	Average	0.3414	0.3771	0.3243
		Std Deviation	0.0703	0.0298	0.1186
SUBSTRATE D	MD	Average	0.6400	0.5929	0.4157
		Std Deviation	0.3749	0.3477	0.1539
	CD	Average	0.8343	0.8286	0.8829
		Std Deviation	0.3090	0.2024	0.2684

Table A.7.1: Dart Drop Chart of Substrate A

Control:

Dart Wt (g)	Sequential drops																					ni	i	ini
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21			
78			X						X		X						X				X	5	1	5
63		O		X		X		O		O		X		X		O		X		O		5	0	0
48	O				O		O						O		O				O					

N: 10

A: 5

Wo = 63g

ΔW = 15g

W_F = 63g

Printed:

Dart Wt (g)	Sequential drops																				ni	i	ini
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20			
93		X																			1	2	2
78	O		X		X		X		X		X		X		X						7	1	7
63				O		O		O		O		O		O		X		X		X	3	0	0
48																	O		O				

N: 10

A: 9

Wo = 63g

ΔW = 15g

W_F = 63g

Table A.7.2: Dart Drop Chart of Substrate B

Control:

Dart Wt	Sequential drops																									ni	i	ini
(g)	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25			
228									X								X								X	3	4	12
213								O		X				X		O		X		X				O		4	3	12
198							O				X		O		O				O		X		O			2	2	4
183						O						O										O				0	1	0
168			X		O																					1	0	0
153		O		O																								
138	O																											

N: 10

A: 28

W_O = 168g

ΔW = 15g

W_F = 202.5g

Printed:

Dart Wt	Sequential drops																						ni	i	ini
(g)	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22			
198									X				X		X				X		X		5	3	15
183								O		X		O		O		X		O		O		X	3	2	6
168					X		O				O						O						1	1	1
153		X		O		O																	1	0	0
138	O		O																						

N: 10

A: 22

W_O = 153g

ΔW = 15g

W_F = 178.5g

Table A.7.3: Dart Drop Chart of Substrate C

Control:

Dart Wt	Sequential drops																				ni	i	ini
(g)	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20			
93		X		X																	2	2	4
78	O		O		X		X		X		X								X		5	1	5
63						O		O		O		X		X		X		O		O	3	0	0
48													O		O		O						

N: 10 **A: 9** **Wo = 63g** **ΔW = 15g** **W_F = 69g**

Printed:

Dart Wt	Sequential drops																				ni	i	ini
(g)	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20			
93															X						1	2	2
78		X		X										O		X					3	1	3
63	O		O		X		X		X		X		O				X		X		6	0	0
48						O		O		O		O						O		O			

N: 10 **A: 5** **Wo = 63g** **ΔW = 15g** **W_F = 63g**

Table A.7.4: Dart Drop Chart of Substrate D

Control:

Dart Wt	Sequential drops																								
(g)	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	ni	i	ini
183				X																			1	3	3
168			O		X		X								X						X		4	2	8
153		O				O		X		X				O		X				O		O	3	1	3
138	O								O		X		O				X		O				2	0	0
123												O						O							

N: 10 **A: 14** **Wo = 138g** **ΔW = 15g** **W_F = 151.5g**

Printed:

Dart Wt	Sequential drops																							
(g)	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	ni	i	ini
198						X																1	3	3
183					O		X						X									2	2	4
168				O				X				O		X								2	1	2
153			O						X		O				X		X		X		X	5	0	0
138		O								O						O		O		O				
123	O																							

N: 10 **A: 9** **Wo = 153g** **ΔW = 15g** **W_F = 159g**

Table A.8.1: Total Migration for Substrate A in 10% ethanol:water simulant

SUSBTRATE A	Initial Wt* (g)	Final Wt* (g)	Total FCS * (mg)	Film thicknesses (m)	Total Migration (mg/dm^2)	
					Average	Std. Deviation
Control - 24 hours	0.5178	0.5165	1.3333	6.096E-05	0.9347	0.5308
Control – 48 hours	0.5072	0.5066	0.6333	6.096E-05	0.4440	0.1071
Control – 120 hours	0.5122	0.5117	0.5000	6.096E-05	0.3505	0.1402
Control – 240 hours	0.5353	0.5344	0.8333	6.096E-05	0.5842	0.1619
Treated – 24 hours	0.5208	0.5199	0.9000	0.0000762	0.6301	0.1852
Treated – 48 hours	0.5206	0.5199	0.7333	0.0000762	0.5134	0.2250
Treated – 120 hours	0.5324	0.5317	0.6333	0.0000762	0.4434	0.1762
Treated – 240 hours	0.5267	0.5258	0.9000	0.0000762	0.6301	0.1852

* All data are the average of 3 replications

Table A.8.2: Total Migration for Substrate B in 10% ethanol:water simulant

SUSBTRATE B	Initial Wt* (g)	Final Wt* (g)	Total FCS * (mg)	Film thicknesses (m)	Total Migration (mg/dm^2)	
					Average	Std. Deviation
Control - 24 hours	0.5838	0.5819	1.8333	0.0000762	1.2835	0.1457
Control – 48 hours	0.5711	0.5697	1.4000	0.0000762	0.9801	0.2524
Control – 120 hours	0.5922	0.5907	1.5333	0.0000762	1.0735	0.1069
Control – 240 hours	0.5838	0.5818	2.0333	0.0000762	1.4235	0.0404
Treated – 24 hours	0.5767	0.5746	2.1000	0.0000762	1.4702	0.0000
Treated – 48 hours	0.5887	0.5876	1.0667	0.0000762	0.7468	0.1069
Treated – 120 hours	0.5942	0.5910	1.3333	0.0000762	0.9335	0.1069
Treated – 240 hours	0.5842	0.5818	2.4667	0.0000762	1.7269	0.1762

* All data are the average of 3 replications

Table A.8.3: Total Migration for Substrate A in 95% ethanol:water simulant

SUSBTRATE A	Initial Wt* (g)	Final Wt* (g)	Total FCS * (mg)	Film thicknesses (m)	Total Migration (mg/dm^2)	
					Average	Std. Deviation
Control - 24 hours	0.5224	0.5202	2.1667	6.096E-05	1.5189	0.8765
Control – 48 hours	0.5273	0.5261	1.2667	6.096E-05	0.8880	0.1459
Control – 120 hours	0.5203	0.5189	1.4000	6.096E-05	0.9814	0.1855
Control – 240 hours	0.5246	0.5231	1.5333	6.096E-05	1.0749	0.1071
Treated – 24 hours	0.5247	0.5237	1.0333	0.0000762	0.7234	0.2021
Treated – 48 hours	0.5302	0.5288	1.3667	0.0000762	0.9568	0.1069
Treated – 120 hours	0.5267	0.5251	1.6000	0.0000762	1.1201	0.1400
Treated – 240 hours	0.5203	0.5185	1.7667	0.0000762	1.2368	0.2021

* All data are the average of 3 replications

Table A.8.4: Total Migration for Substrate B in 95% ethanol:water simulant

SUSBTRATE B	Initial Wt* (g)	Final Wt* (g)	Total FCS * (mg)	Film thicknesses (m)	Total Migration (mg/dm^2)	
					Average	Std. Deviation
Control - 24 hours	0.5914	0.5868	4.6000	0.0000762	3.2204	1.2660
Control – 48 hours	0.5941	0.5916	1.0639	0.0000762	1.7036	0.0808
Control – 120 hours	0.5799	0.5771	2.8333	0.0000762	1.9836	0.2021
Control – 240 hours	0.5844	0.5811	3.3000	0.0000762	2.3103	0.3898
Treated – 24 hours	0.6023	0.5990	3.3667	0.0000762	2.3570	1.1252
Treated – 48 hours	0.5797	0.5775	2.2000	0.0000762	1.5402	0.6223
Treated – 120 hours	0.5756	0.5735	2.1333	0.0000762	1.4935	0.2829
Treated – 240 hours	0.5705	0.5670	-3.5000	0.0000762	2.4503	0.2100

* All data are the average of 3 replications

Table A.8.5: Total FCS for Substrate C in 10% ethanol:water simulant

SUSBTRATE C	Initial Wt* (g)	Final Wt* (g)	Total FCS (mg)
Control - 24 hours	0.0669	0.0668	-0.10
Control – 48 hours	0.0655	0.0654	-0.10
Control – 120 hours	0.0660	0.0667	0.70
Control – 240 hours	0.0654	0.0657	0.30
Treated – 24 hours	0.0799	0.0794	-0.50
Treated – 48 hours	0.0803	0.0798	-0.50
Treated – 120 hours	0.0822	0.0816	-0.60
Treated – 240 hours	0.0809	0.0801	-0.80

Table A.8.6: Total FCS for Substrate D in 10% ethanol:water simulant

SUSBTRATE C	Initial Wt* (g)	Final Wt* (g)	Total FCS (mg)
Control - 24 hours	0.0954	0.0954	0.00
Control – 48 hours	0.0925	0.0922	-0.30
Control – 120 hours	0.0952	0.0952	0.00
Control – 240 hours	0.0944	0.0941	-0.30
Treated – 24 hours	0.1088	0.1082	-0.60
Treated – 48 hours	0.1077	0.1072	-0.50
Treated – 120 hours	0.1085	0.1080	-0.50
Treated – 240 hours	0.1078	0.1083	0.50

Table A.8.7: Total FCS for Substrate C in 95% ethanol:water simulant

SUSBTRATE C	Initial Wt (g)	Final Wt (g)	Total FCS (mg)
Control - 24 hours	0.0652	0.0653	0.10
Control – 48 hours	0.0669	0.0671	0.20
Control – 120 hours	0.0667	0.0663	-0.40
Control – 240 hours	0.0683	0.0751	6.80
Treated – 24 hours	0.0845	0.0807	-3.80
Treated – 48 hours	0.0851	0.0841	-1.00
Treated – 120 hours	0.0813	0.0805	-0.80
Treated – 240 hours	0.0848	0.0840	-0.80

Table A.8.8: Total FCS for Substrate D in 95% ethanol:water simulant

SUSBTRATE C	Initial Wt (g)	Final Wt (g)	Total FCS (mg)
Control - 24 hours	0.0951	0.0949	-0.20
Control – 48 hours	0.0931	0.0929	-0.20
Control – 120 hours	0.0902	0.0899	-0.30
Control – 240 hours	0.0955	0.0951	-0.40
Treated – 24 hours	0.1032	0.1029	-0.30
Treated – 48 hours	0.1092	0.1084	-0.80
Treated – 120 hours	0.1096	0.1089	-0.70
Treated – 240 hours	0.1067	0.1056	-1.10

APPENDIX B

STATISTICAL ANALYSIS AND CALCULATION EXAMPLE

STATISTICAL ANALYSIS OF TENSILE STRENGTH:

```
                            The GLM Procedure
                           Least Squares Means
           materi*treat*directi Effect Sliced by material for ts

      material    DF    Sum of Squares    Mean Square   F Value   Pr > F
         1         7        1443432026      206204575    115.28   <.0001
         2         7            440485          62926      0.04   0.9999
         3         7        4001103311      571586187    319.54   <.0001
         4         7        7570903924     1081557703    604.63   <.0001


        materi*treat*directi Effect Sliced by material*treat for ts

material   treat     DF  Sum of Squares   Mean Square        F Value        Pr > F
   1       ctrl       1     459531463      459531463          256.89        <.0001
   1       uncoat     1     398504860      398504860          222.78        <.0001
   1       coat       1     287553338      287553338          160.75        <.0001
   1       printed    1     265205065      265205065          148.26        <.0001
   2       ctrl       1   7182.400054    7182.400054            0.00        0.9496
   2       uncoat     1         77206          77206            0.04        0.8358
   2       coat       1         20976          20976            0.01        0.9139
   2       printed    1   1181.779409    1181.779409            0.00        0.9795
   3       ctrl       1    1184427985     1184427985          662.14        <.0001
   3       uncoat     1    1253168303     1253168303          700.57        <.0001
   3       coat       1     786262225      786262225          439.55        <.0001
   3       printed    1     640800250      640800250          358.23        <.0001
   4       ctrl       1    2213548840     2213548840         1237.45        <.0001
   4       uncoat     1    1731066490     1731066490          967.73        <.0001
   4       coat       1    1672789496     1672789496          935.15        <.0001
   4       printed    1    1851826123     1851826123         1035.24        <.0001


      materi*treat*directi Effect Sliced by material*direction for ts

  material   direction    DF   Sum of Squares   Mean Square   F Value      Pr > F
     1          m          3        49343251       16447750      9.19      <.0001
     1          c          3         1239680         413227      0.23      0.8747
     2          m          3          275353          91784      0.05      0.9846
     2          c          3          156722          52241      0.03      0.9932
     3          m          3        34167446       11389149      6.37      0.0005
     3          c          3       174221346       58073782     32.47      <.0001
     4          m          3        14426216        4808739      2.69      0.0492
     4          c          3       109905001       36635000     20.48      <.0001
```

STATISTICAL ANALYSIS OF THE PEAK LOAD:

```
                          The GLM Procedure
                         Least Squares Means

Source                DF   Type III SS   Mean Square   F Value   Pr > F

material               3   12203.73428    4067.91143   2312.53   <.0001
direction              1     188.70770     188.70770    107.28   <.0001
material*direction     3   20913.19858    6971.06619   3962.92   <.0001
treat                  3       9.52146       3.17382      1.80   0.1497
material*treat         9     137.94372      15.32708      8.71   <.0001
direction*treat        3      34.30347      11.43449      6.50   0.0004
materi*directi*treat   9      35.96887       3.99654      2.27   0.0214
```

```
        material*direction Effect Sliced by material for tspeak

   material     DF     Sum of Squares   Mean Square    F Value   Pr > F

      A          1     11098            11098          6309.10   <.0001
      B          1        0.074736         0.074736       0.04   0.8370
      C          1     1815.203817      1815.203817    1031.91   <.0001
      D          1     8188.468402      8188.468402    4654.99   <.0001
```

```
         material*treat Effect Sliced by material for tspeak

                       Sum of
   material     DF     Squares    Mean Square   F Value    Pr > F

      A          3    46.583433    15.527811     8.83      <.0001
      B          3     1.634461     0.544820     0.31      0.8183
      C          3    12.996159     4.332053     2.46      0.0655
      D          3    86.251127    28.750376    16.34      <.0001
```

STATISTICAL ANALYSIS OF ELMENDORF TEAR STRENGTH:

```
                              full model
                          The GLM Procedure
Dependent Variable: lntear

Source            DF    Sum of Squares     Mean Square  F Value   Pr > F
Model              23   766.1460056        33.3106959   3751.81   <.0001
Error             216     1.9177698         0.0088786
Corrected Total   239   768.0637755

                  R-Square   Coeff Var   Root MSE   lntear Mean
                  0.997503   2.778681    0.094226    3.391038

Source                DF  Type III SS   Mean Square       F Value   Pr > F

material               3  708.8706144   236.2902048       26613.6   <.0001
direction              1    9.1312243     9.1312243       1028.46   <.0001
material*direction     3   45.6164981    15.2054994       1712.61   <.0001
treat                  2    0.2303226     0.1151613       12.97     <.0001
material*treat         6    1.3903245     0.2317207        26.10    <.0001
direction*treat        2    0.0234593     0.0117296         1.32    0.2690
materi*directi*treat   6    0.8835626     0.1472604        16.59    <.0001


               material*treat Effect Sliced by material for lntear

  material    DF    Sum of Squares   Mean Square   F Value   Pr > F

     A         2       0.072989       0.036494        4.11   0.0177
     B         2       0.256844       0.128422       14.46   <.0001
     C         2       0.062267       0.031133        3.51   0.0317
     D         2       1.228548       0.614274       69.19   <.0001

           material*direction Effect Sliced by material for lntear

  material    DF    Sum of Squares   Mean Square   F Value   Pr > F

     A         1      11.956741      11.956741    1346.70   <.0001
     B         1      36.779929      36.779929    4142.55   <.0001
     C         1       2.216968       2.216968     249.70   <.0001
     D         1       3.794084       3.794084     427.33   <.0001
```

STATISTICAL ANALYSIS OF PEAK SEAL STRENGTH:

```
                                full model
                            The GLM Procedure
Dependent Variable: peak
Source              DF     Sum of Squares   Mean Square   F Value   Pr > F
Model               23        259.3084511    11.2742805    190.80   <.0001
Error              144          8.5087974     0.0590889
Corrected Total    167        267.8172485

                R-Square    Coeff Var    Root MSE    peak Mean
                0.968229     19.74518    0.243082     1.231095

Source                DF     Type III SS   Mean Square   F Value   Pr > F

material               3     251.4488633    83.8162878   1418.48   <.0001
direction              1       1.0867481     1.0867481     18.39   <.0001
material*direction     3       3.6829840     1.2276613     20.78   <.0001
treat                  2       0.0309656     0.0154828      0.26   0.7699
material*treat         6       1.8283756     0.3047293      5.16   <.0001
direction*treat        2       0.0960317     0.0480159      0.81   0.4457
materi*directi*treat   6       1.1344827     0.1890805      3.20   0.0056
```

```
             material*direction Effect Sliced by material for peak

     material     DF     Sum of Squares   Mean Square   F Value   Pr > F

        A          1           3.386124      3.386124     57.31   <.0001
        B          1           0.059890      0.059890      1.01   0.3157
        C          1           0.661921      0.661921     11.20   0.0010
        D          1           0.661796      0.661796     11.20   0.0010
```

```
             material*treat Effect Sliced by material for peak

     material     DF     Sum of Squares   Mean Square   F Value   Pr > F

        A          2           0.794424      0.397212      6.72   0.0016
        B          2           0.024503      0.012252      0.21   0.8130
        C          2           0.748228      0.374114      6.33   0.0023
        D          2           0.292186      0.146093      2.47   0.0880
```

DART DROP CALCULATION EXAMPLE:

SUBSTRATE A: CONTROL FILM

Dart Wt (g)	Sequential drops																					ni	i	ini
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21			
78			X						X		X						X				X	5	1	5
63		O		X		X		O		O		X		X		O		X		O		5	0	0
48	O				O		O						O		O				O					

N: 10
A: 5
Wo = 63g
ΔW = 15g
W_F = 63g

$$W_F = W_O + \left[\Delta W\left(\frac{A}{N} - \frac{1}{2}\right)\right]$$

$$W_F = 63 + \left[15\left(\frac{5}{10} - \frac{1}{2}\right)\right] = \underline{\mathbf{63\ g}}$$

STATISTICAL ANALYSIS OF TOTAL MIGRATION :

Total Migration in 10% ethanol:water simulant

```
                                full model
                            The GLM Procedure
Dependent Variable: mig
   Source              DF      Sum of Squares   Mean Square   F Value     Pr > F
   Model               15          7.80693490    0.52046233     12.98     <.0001
   Error               32          1.28279827    0.04008745
   Corrected Total     47          9.08973317

            R-Square    Coeff Var    Root MSE    mig Mean
            0.858874    22.61036     0.200218    0.885517

   Source                    DF   Type III SS    Mean Square   F Value    Pr > F

   material                   1   4.89142083     4.89142083    122.02     <.0001
   films                      1   0.00007752     0.00007752      0.00     0.9652
   material*films             1   0.00850669     0.00850669      0.21     0.6482
   hours                      3   1.92365358     0.64121786     16.00     <.0001
   material*hours             3   0.52791051     0.17597017      4.39     0.0107
   films*hours                3   0.12367742     0.04122581      1.03     0.3931
   material*films*hours       3   0.33168835     0.11056278      2.76     0.0583

                 material*films Effect Sliced by material for mig

      material    DF      Sum of Squares   Mean Square   F Value    Pr > F
      A10         1             0.003480      0.003480      0.09    0.7702
      B10         1             0.005104      0.005104      0.13    0.7236

                  films*hours Effect Sliced by hours for mig

      hours      DF       Sum of Squares   Mean Square   F Value    Pr > F
       24        1              0.010425      0.010425      0.26    0.6136
       48        1              0.020156      0.020156      0.50    0.4834
      120        1              0.001666      0.001666      0.04    0.8397
      240        1              0.091508      0.091508      2.28    0.1406


                 material*hours Effect Sliced by material for mig

      material    DF      Sum of Squares   Mean Square   F Value    Pr > F
      A10         3             0.508336      0.169445      4.23    0.0126
      B10         3             1.943228      0.647743     16.16    <.0001
```

Total Migration in 95% ethanol:water simulant

```
                              full model
                          The GLM Procedure
Dependent Variable: lnmig

   Source             DF     Sum of Squares   Mean Square   F Value    Pr > F
   Model              15         7.87143470    0.52476231      6.97    <.0001
   Error              32         2.40966036    0.07530189
   Corrected Total    47        10.28109506

          R-Square    Coeff Var    Root MSE    lnmig Mean
          0.765622    76.70448     0.274412    0.357752

   Source                    DF    Type III SS    Mean Square   F Value   Pr > F

   material                  1      5.56806069     5.56806069     73.94   <.0001
   films                     1      0.20040213     0.20040213      2.66   0.1126
   material*films            1      0.02988511     0.02988511      0.40   0.5332
   hours                     3      0.81408388     0.27136129      3.60   0.0238
   material*hours            3      0.41745037     0.13915012      1.85   0.1584
   films*hours               3      0.59419769     0.19806590      2.63   0.0670
   material*films*hours      3      0.24735484     0.08245161      1.09   0.3655


              material*films Effect Sliced by material for lnmig

      material    DF     Sum of Squares    Mean Square   F Value    Pr > F
      A95         1            0.037755       0.037755      0.50    0.4840
      B95         1            0.192533       0.192533      2.56    0.1196


              material*hours Effect Sliced by material for lnmig

      material    DF     Sum of Squares    Mean Square   F Value    Pr > F
      A95         3            0.162592       0.054197      0.72    0.5476
      B95         3            1.068943       0.356314      4.73    0.0076


                films*hours Effect Sliced by films for lnmig

      films       DF     Sum of Squares    Mean Square   F Value    Pr > F
      control     3            0.859254       0.286418      3.80    0.0194
      treated     3            0.549028       0.183009      2.43    0.0833
```

APPENDIX C

UV/VIS SPECTRUMS

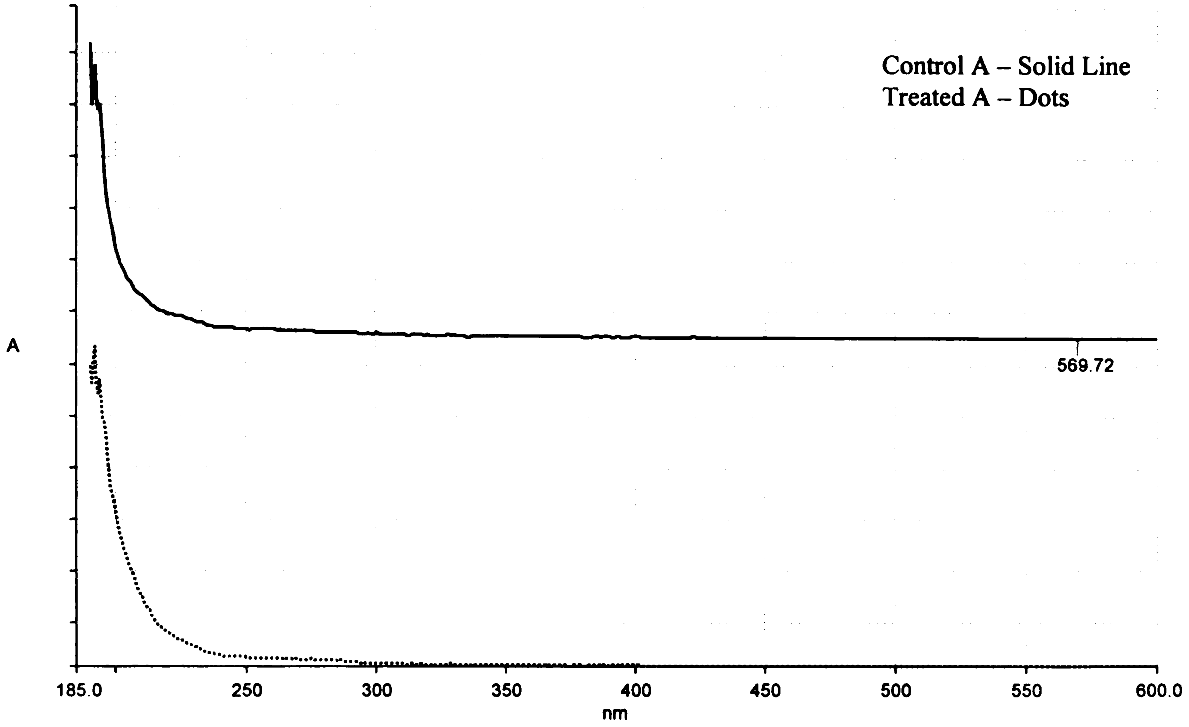


Figure C.1.1: Substrate A in 10% Ethanol for 24 hours

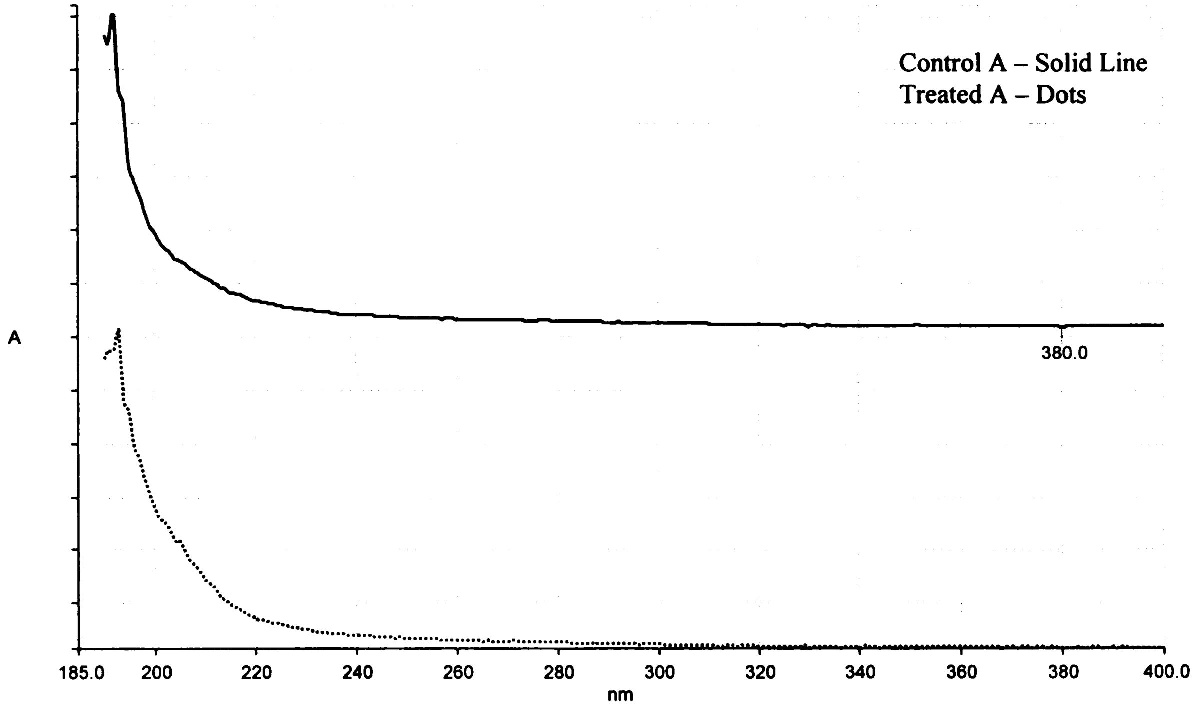


Figure C.1.2: Substrate A in 10% Ethanol for 48 hours

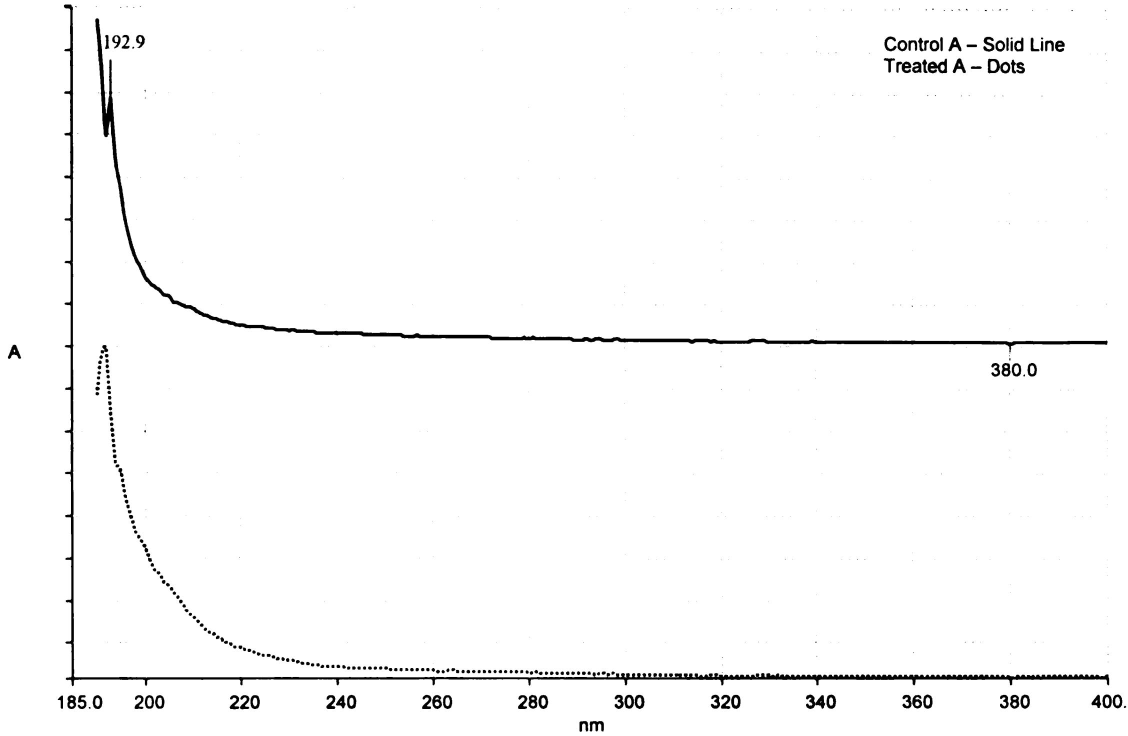


Figure C.1.3: Substrate A in 10% Ethanol for 120 hours

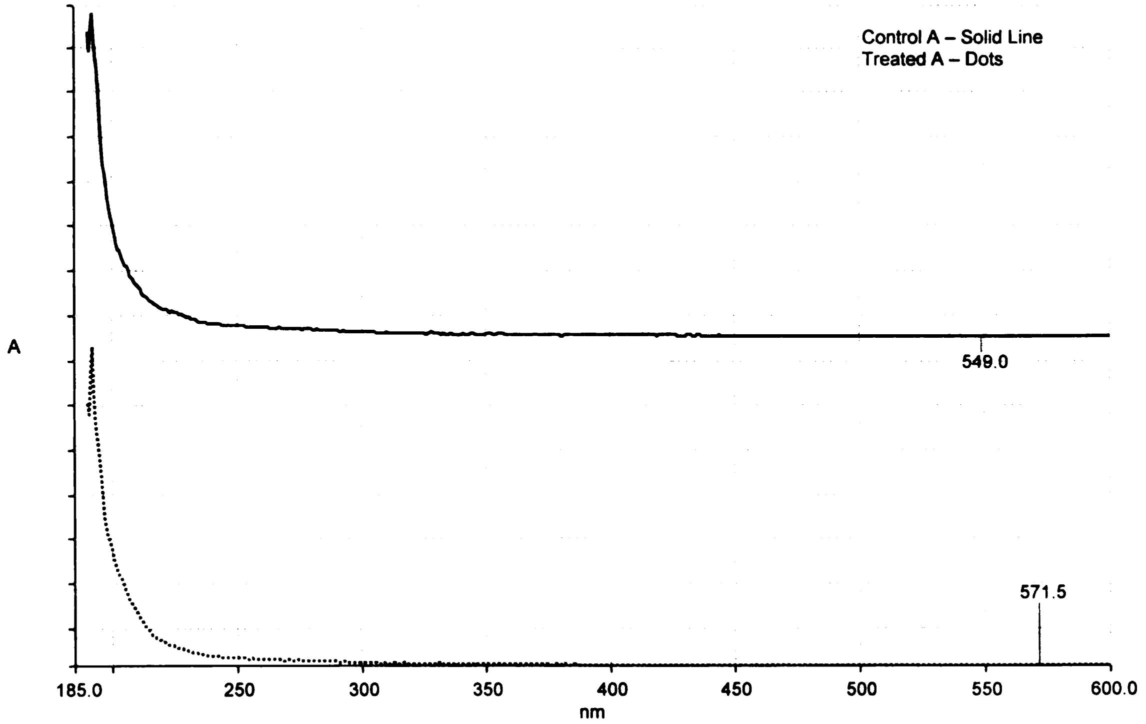


Figure C.1.4: Substrate A in 10% Ethanol for 240 hours

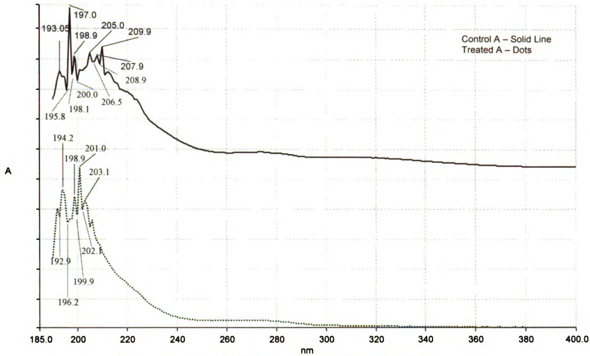


Figure C.1.5: Substrate A in 95% Ethanol for 24 hours

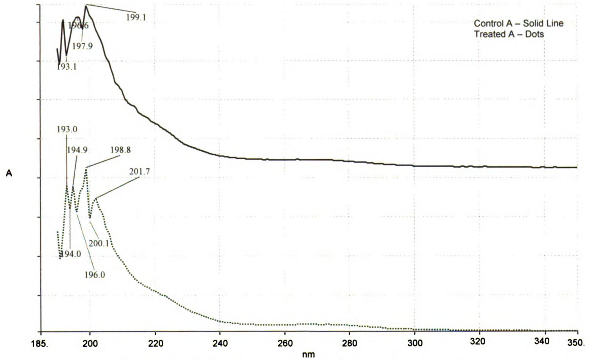


Figure C.1.6: Substrate A in 95% Ethanol for 48 hours

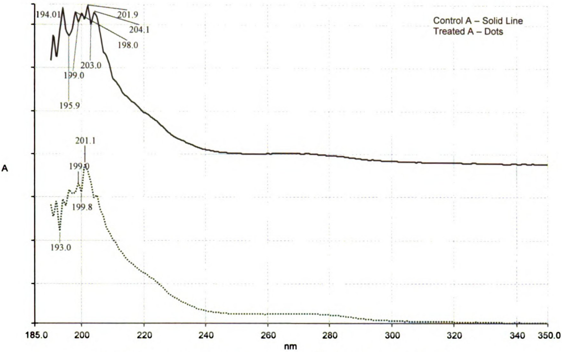


Figure C.1.7: Substrate A in 95% Ethanol for 120 hours

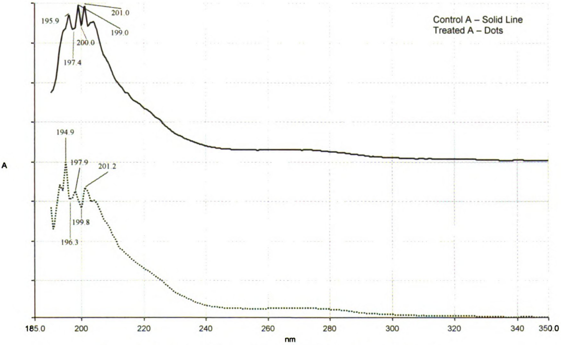


Figure C.1.8: Substrate A in 95% Ethanol for 240 hours

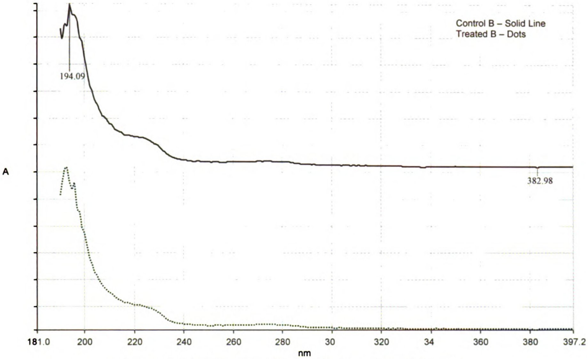


Figure C.2.1: Substrate B in 10% Ethanol for 24 hours

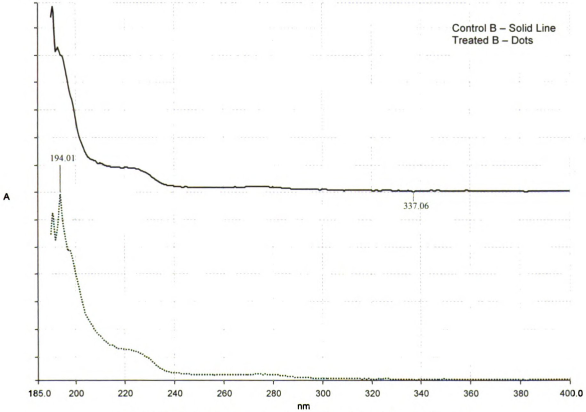


Figure C.2.2: Substrate B in 10% Ethanol for 48 hours

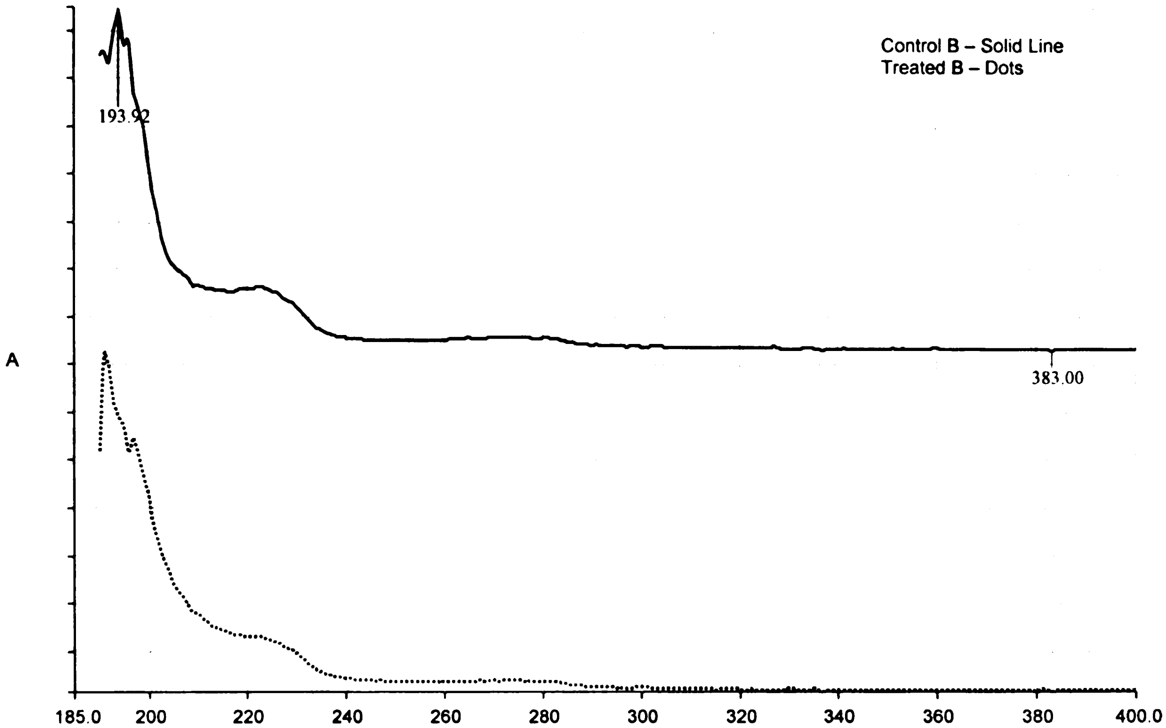


Figure C.2.3: Substrate B in 10% Ethanol for 120 hours

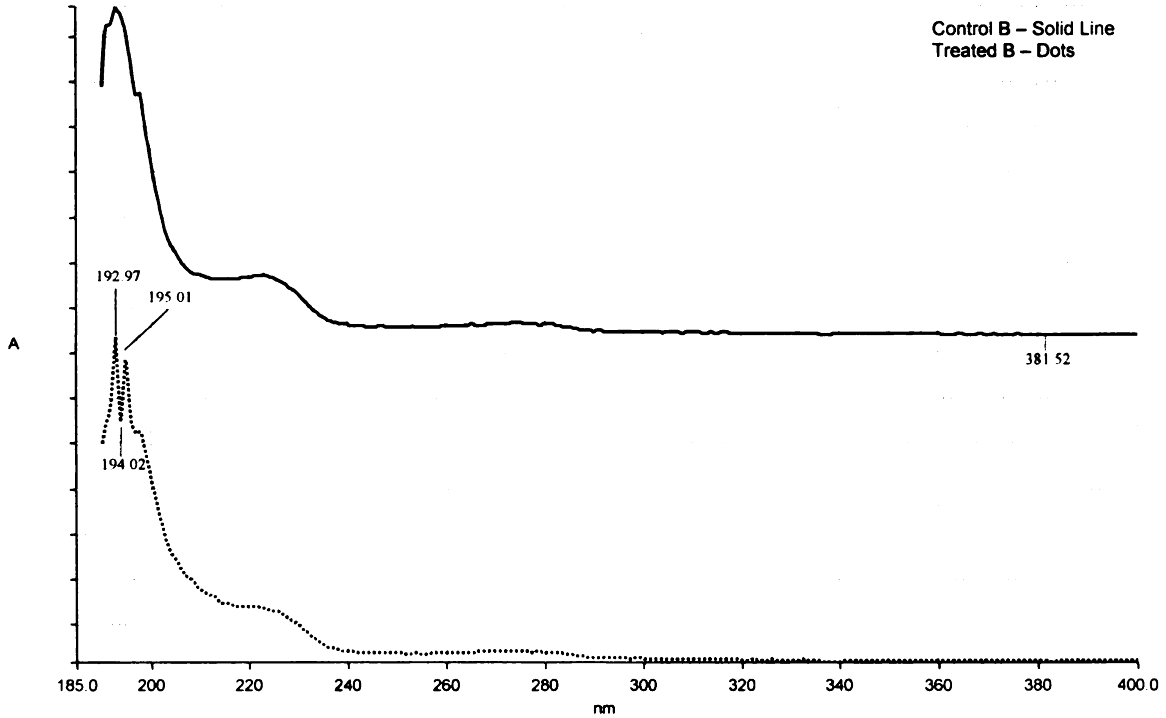


Figure C.2.4: Substrate B in 10% Ethanol for 240 hours

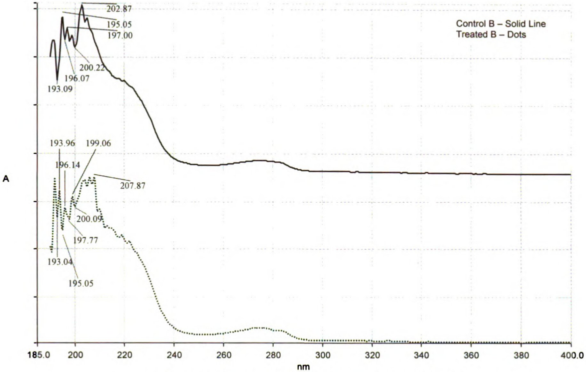


Figure C.2.5: Substrate B in 95% Ethanol for 24 hours

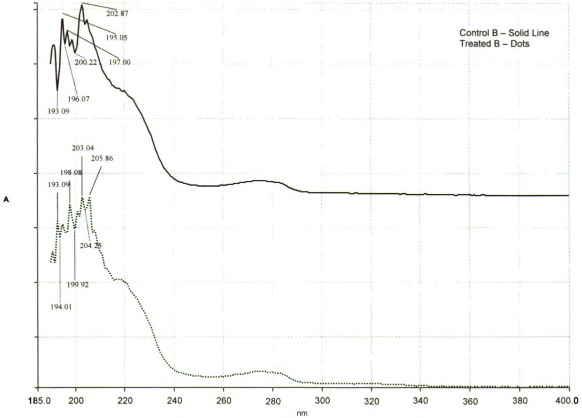


Figure C.2.6: Substrate B in 95% Ethanol for 48 hours

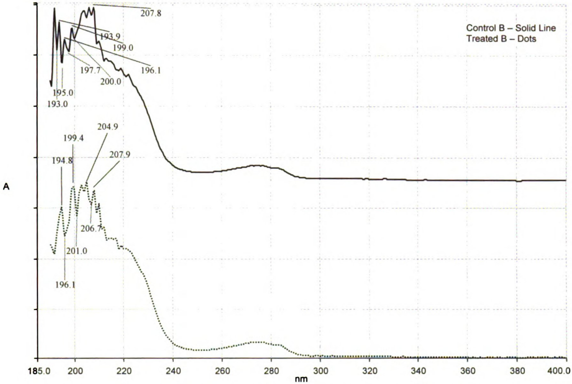


Figure C.2.7: Substrate B in 95% Ethanol for 120 hours

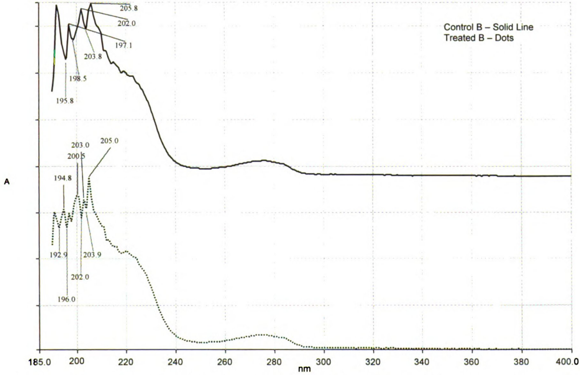


Figure C.2.8: Substrate B in 95% Ethanol for 240 hours

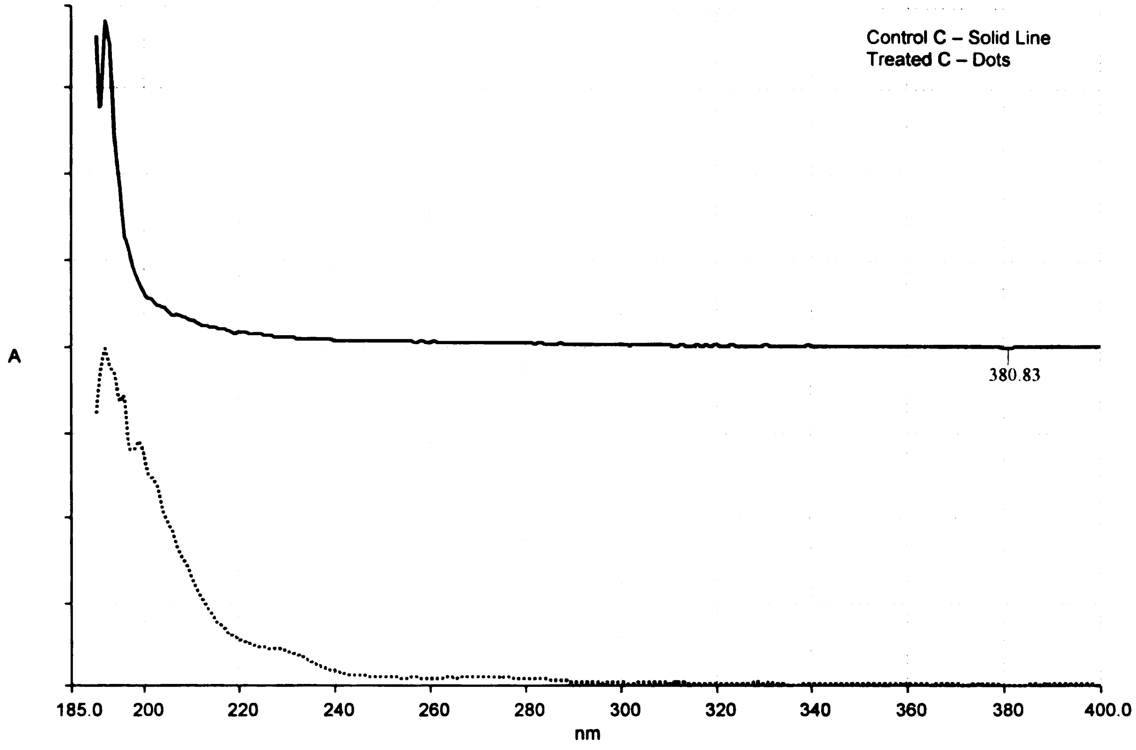


Figure C.3.1: Substrate C in 10% Ethanol for 24 hours

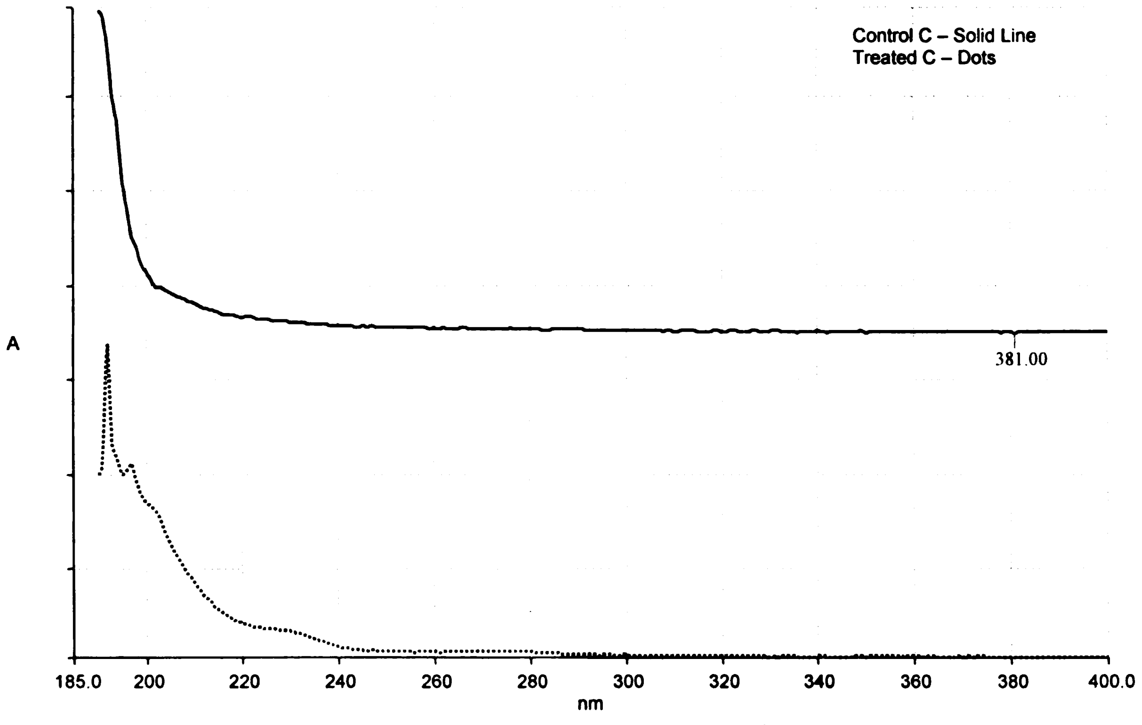


Figure C.3.2: Substrate C in 10% Ethanol for 48 hours

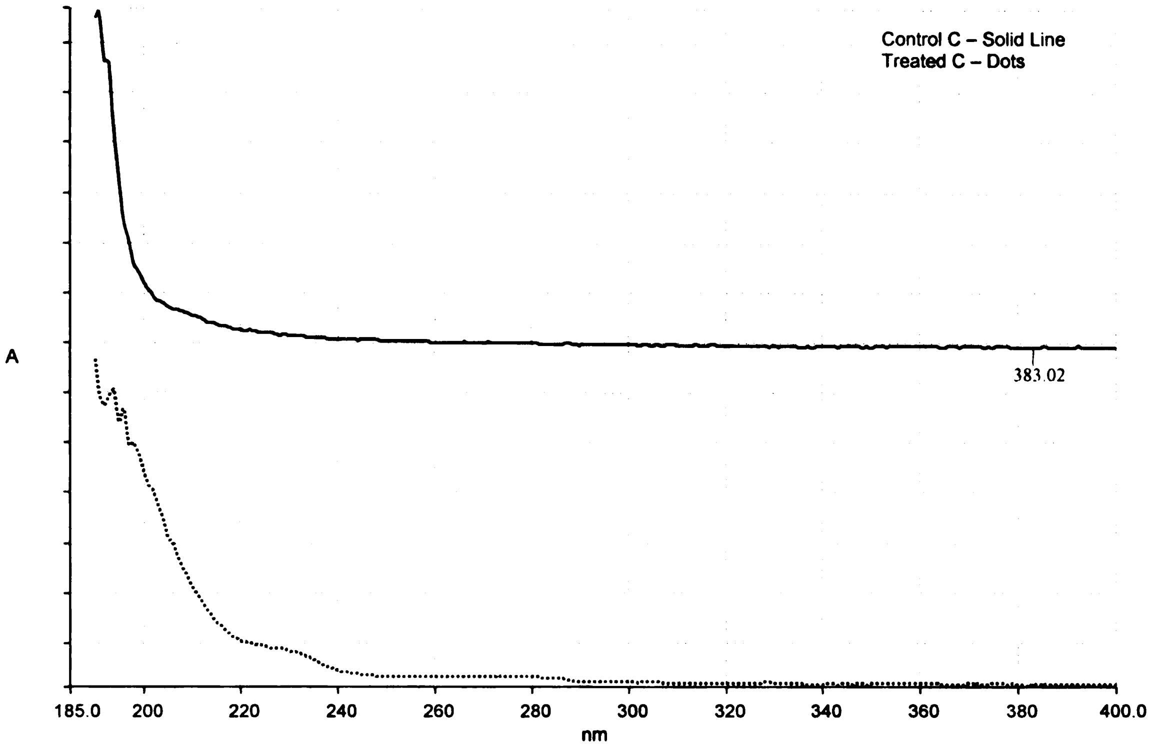


Figure C.3.3: Substrate C in 10% Ethanol for 120 hours

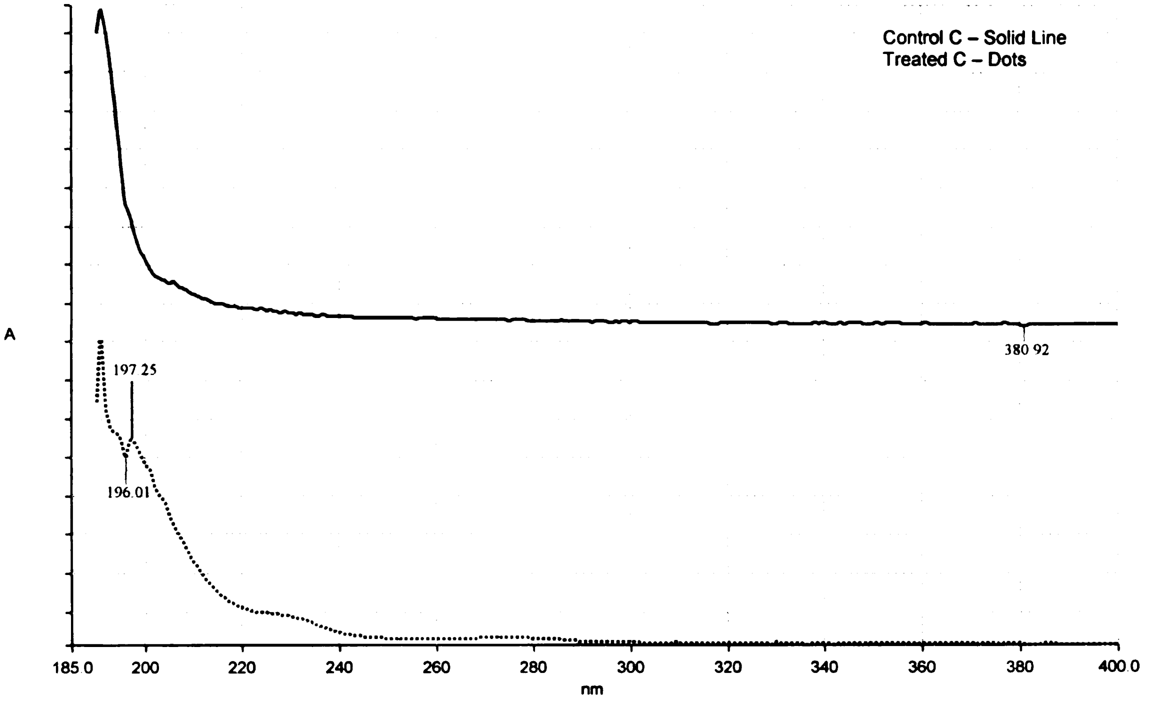


Figure C.3.4: Substrate C in 10% Ethanol for 240 hours

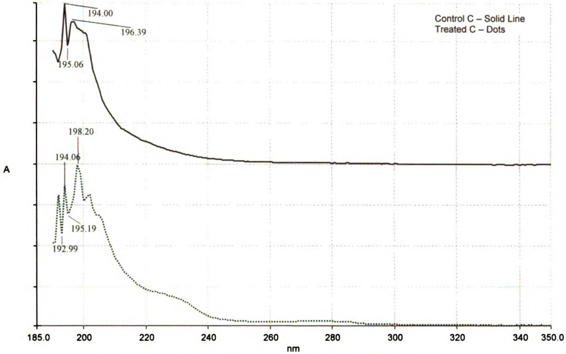


Figure C.3.5: Substrate C in 95% Ethanol for 24 hours

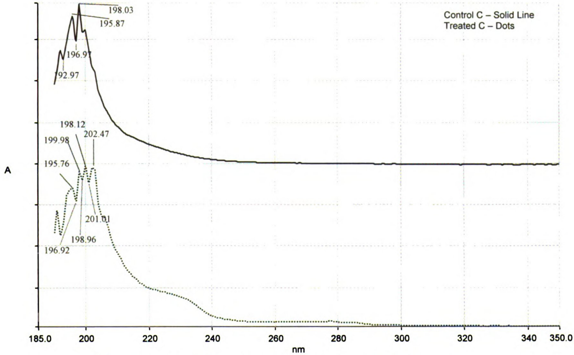


Figure C.3.6: Substrate C in 95% Ethanol for 48 hours

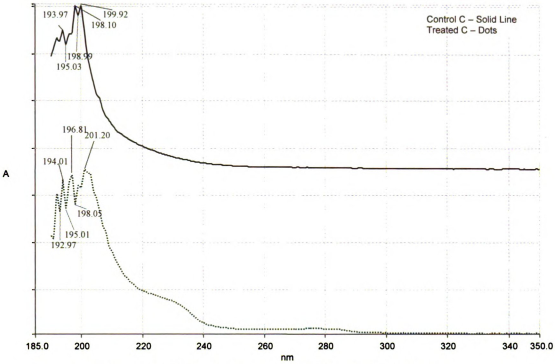


Figure C.3.7: Substrate C in 95% Ethanol for 120 hours

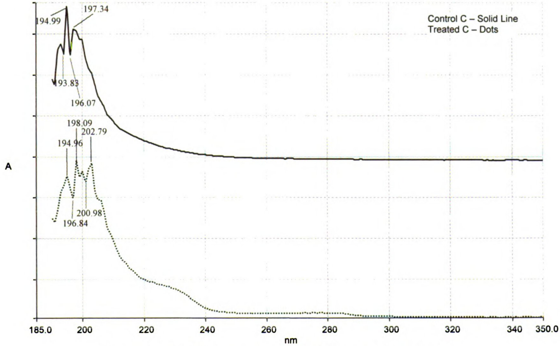


Figure C.3.8: Substrate C in 95% Ethanol for 240 hours

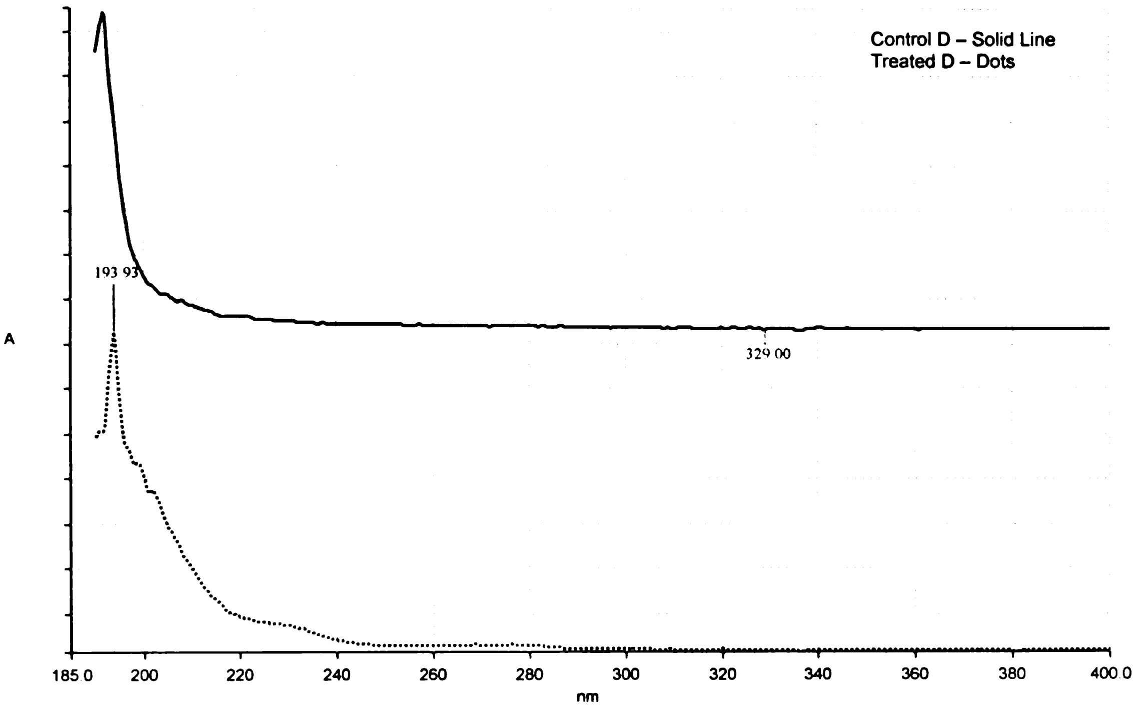


Figure C.4.1: Substrate D in 10% Ethanol for 24 hours

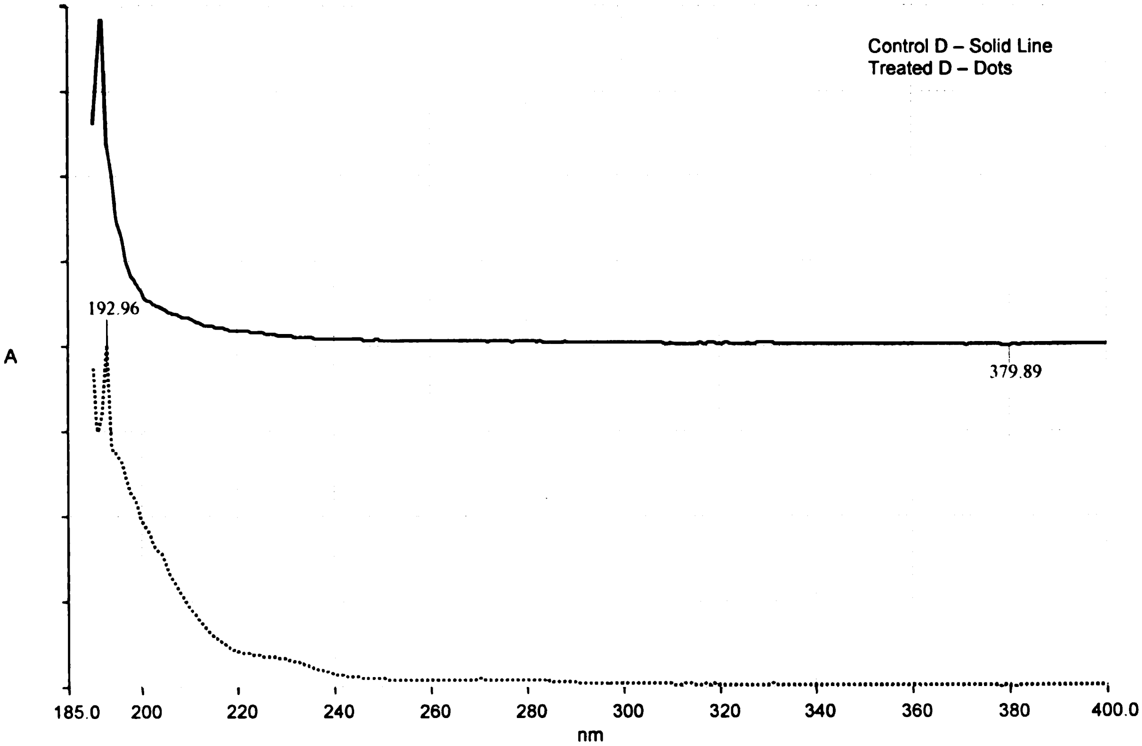


Figure C.4.2: Substrate D in 10% Ethanol for 48 hours

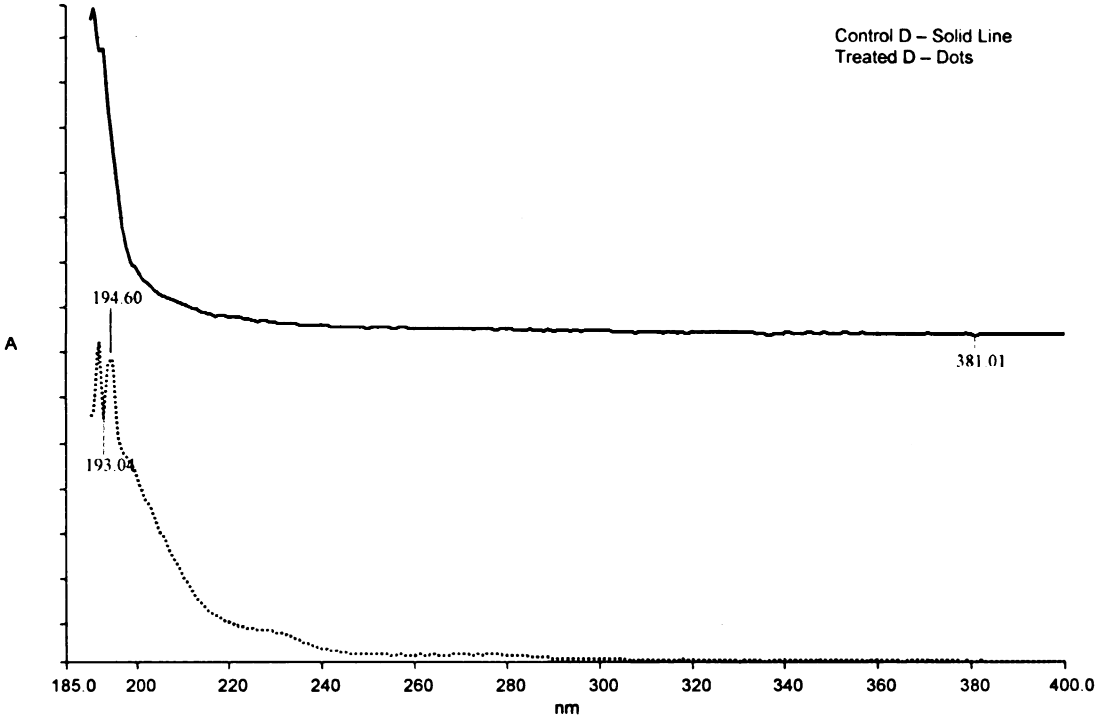


Figure C.4.3: Substrate D in 10% Ethanol for 120 hours

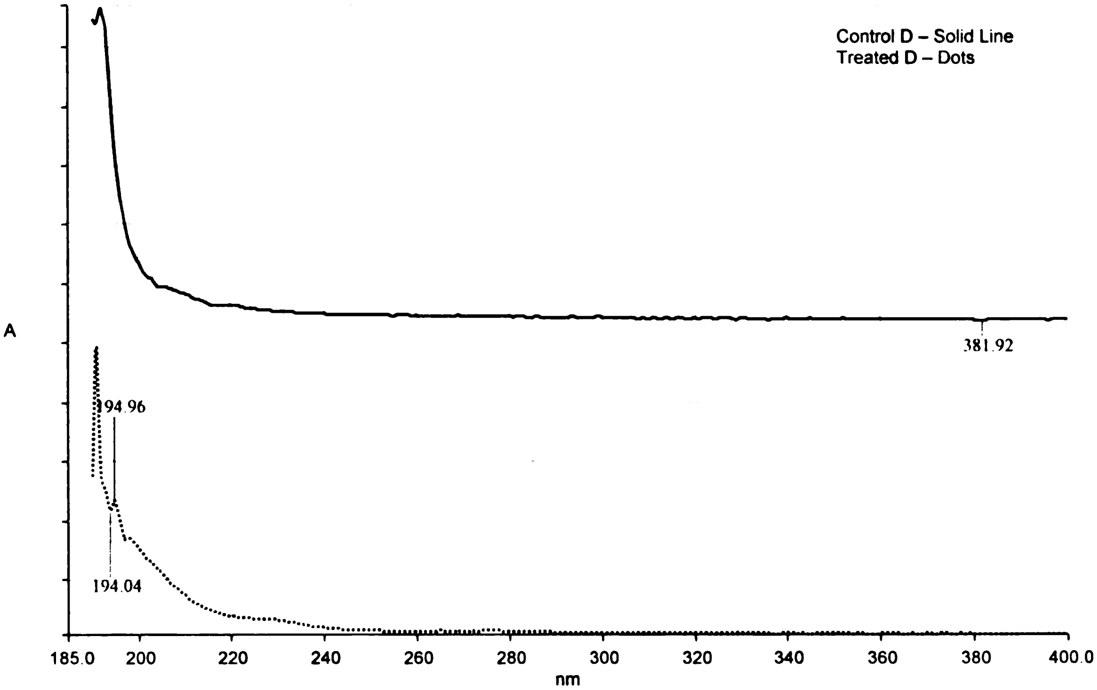


Figure C.4.4: Substrate D in 10% Ethanol for 240 hours

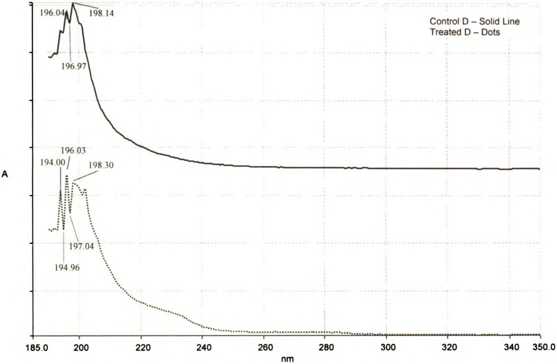


Figure C.4.5: Substrate D in 95% Ethanol for 24 hours

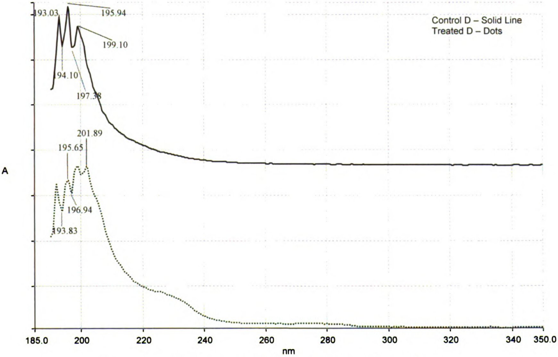


Figure C.4.6: Substrate D in 95% Ethanol for 48 hours

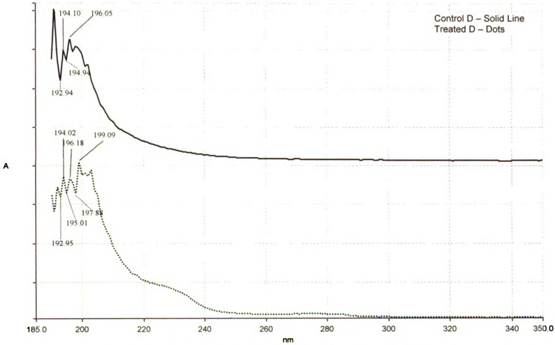


Figure C.4.7: Substrate D in 95% Ethanol for 120 hours

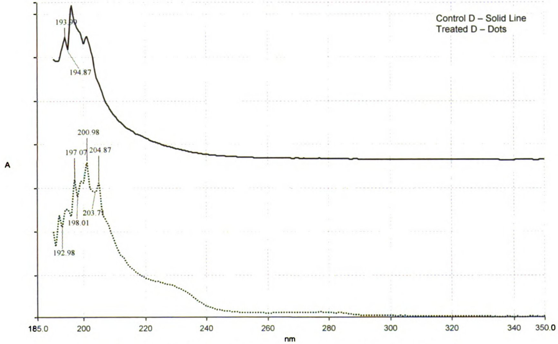


Figure C.4.8: Substrate D in 95% Ethanol for 240 hours

APPENDIX D

FTIR SPECTRUMS

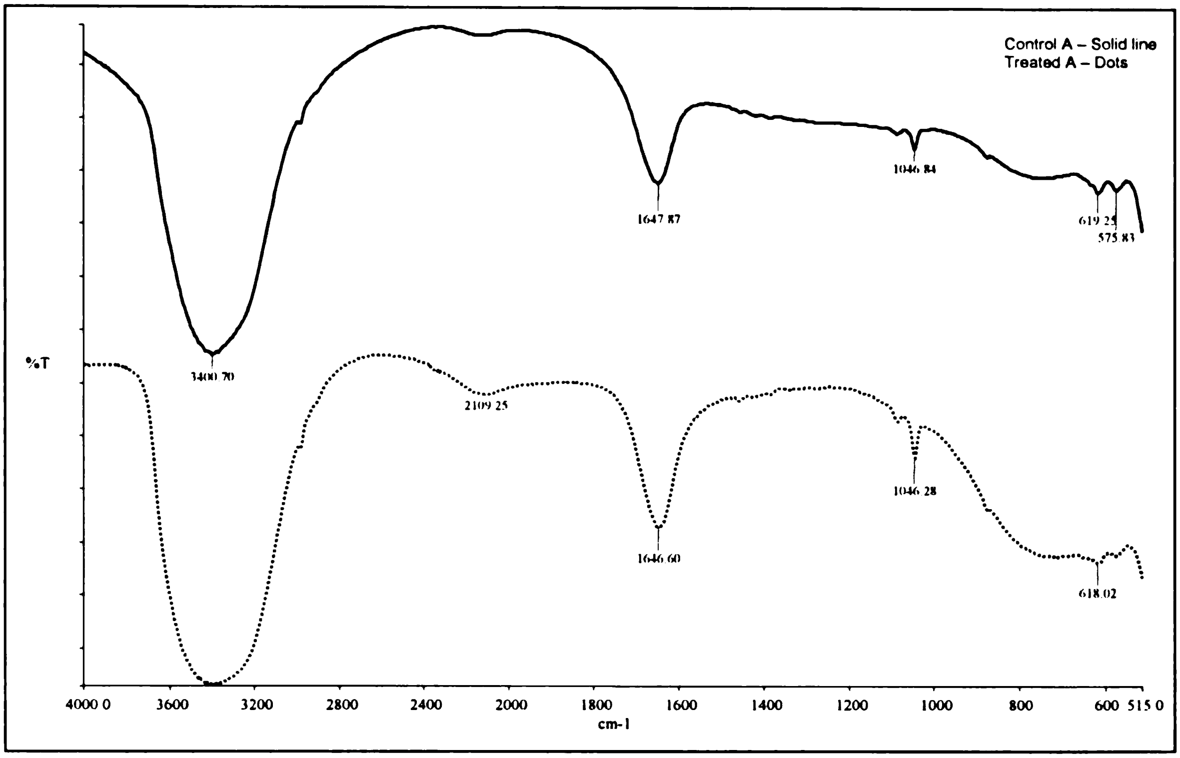


Figure D.1.1: Percent Transmittance of Substrate A in 10% Ethanol for 24 hours

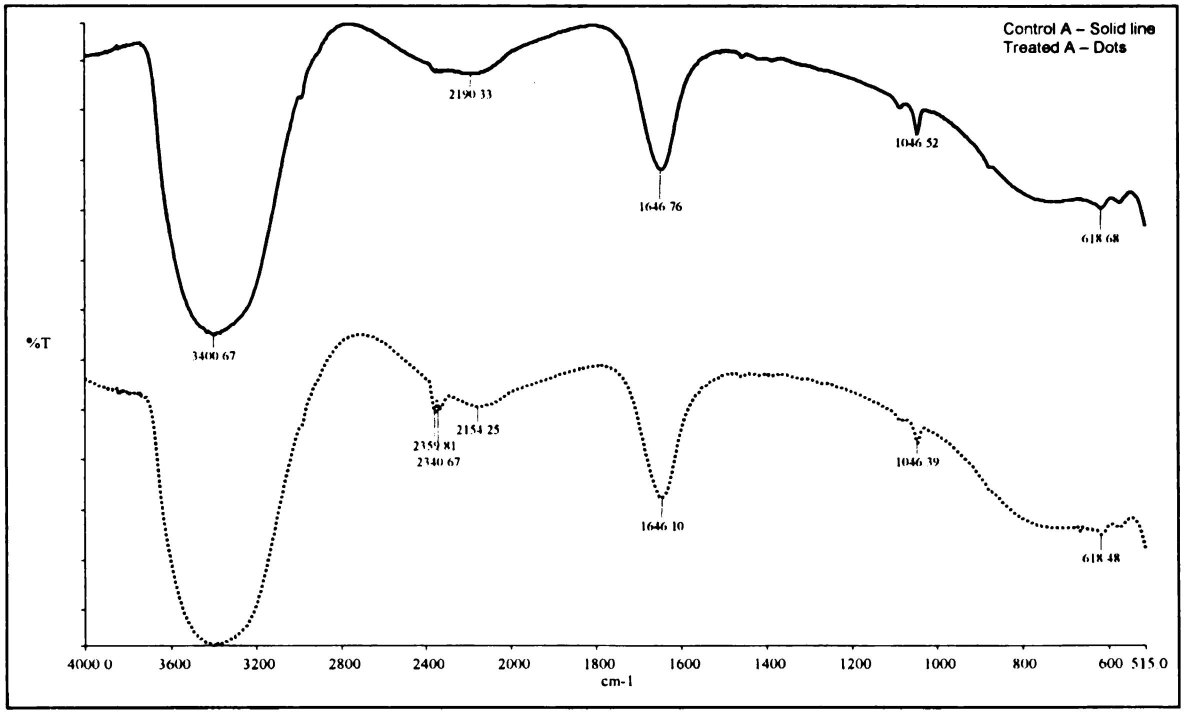


Figure D.1.2: Percent Transmittance of Substrate A in 10% Ethanol for 48 hours

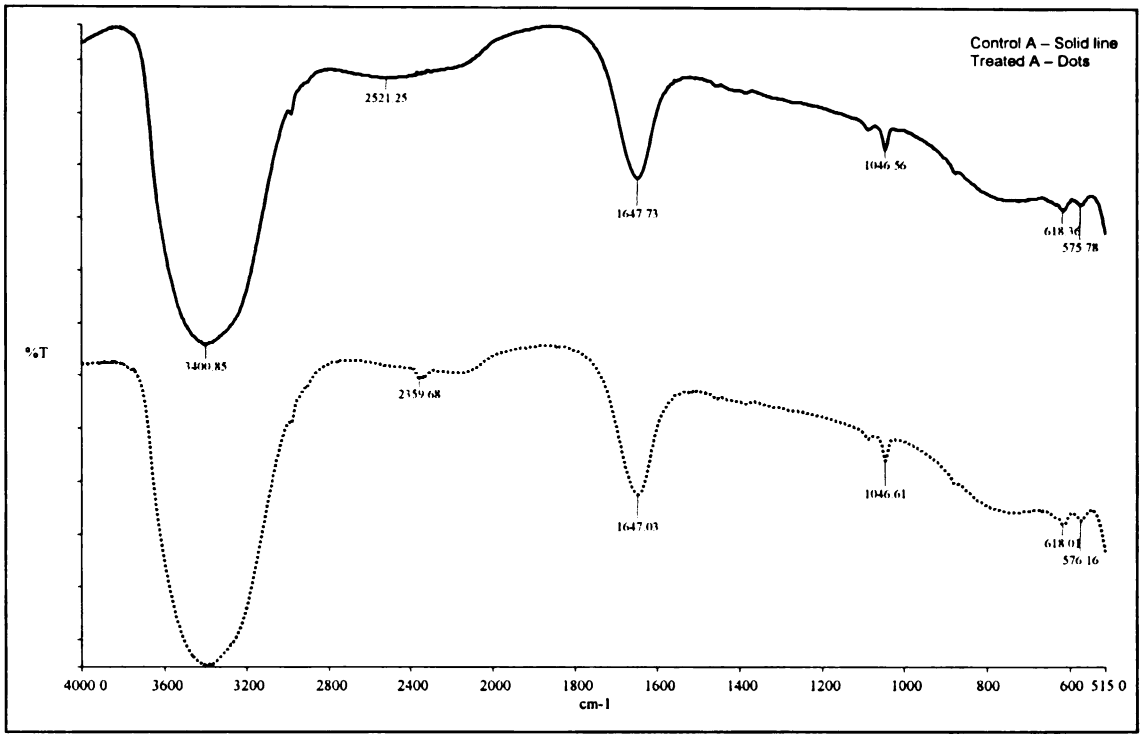


Figure D.1.3: Percent Transmittance of Substrate A in 10% Ethanol for 120 hours

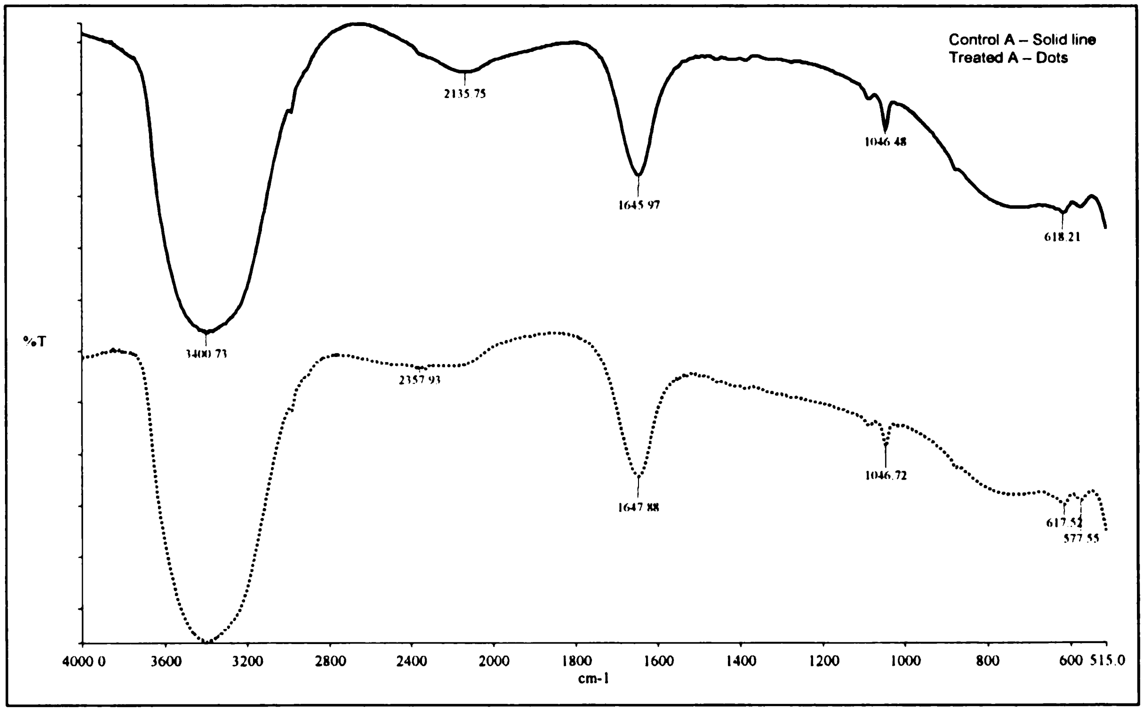


Figure D.1.4: Percent Transmittance of Substrate A in 10% Ethanol for 240 hours

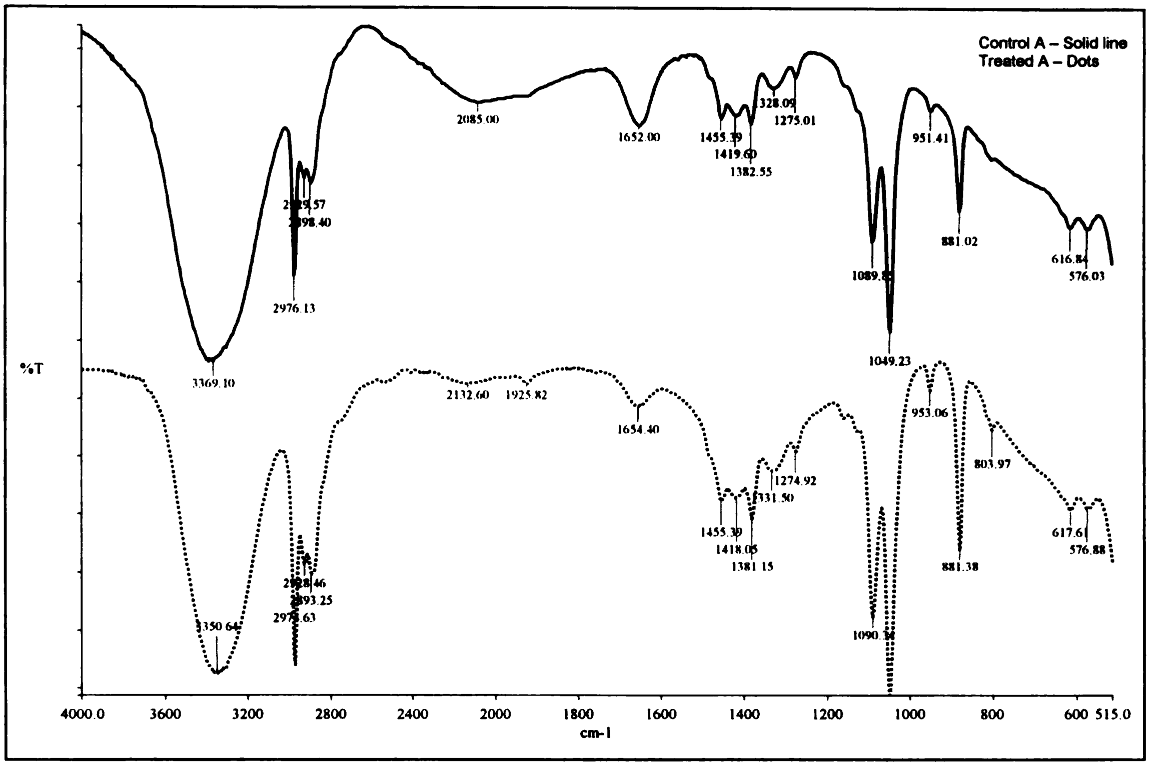


Figure D.1.5: Percent Transmittance of Substrate A in 95% Ethanol for 24 hours

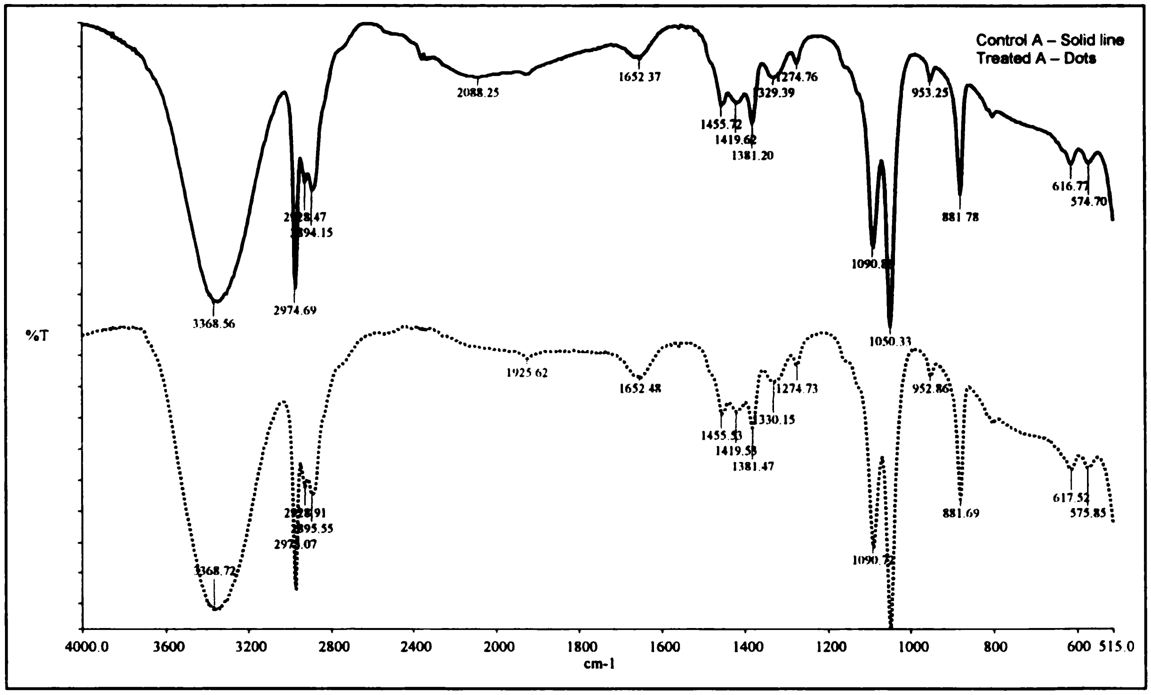


Figure D.1.6: Percent Transmittance of Substrate A in 95% Ethanol for 48 hours

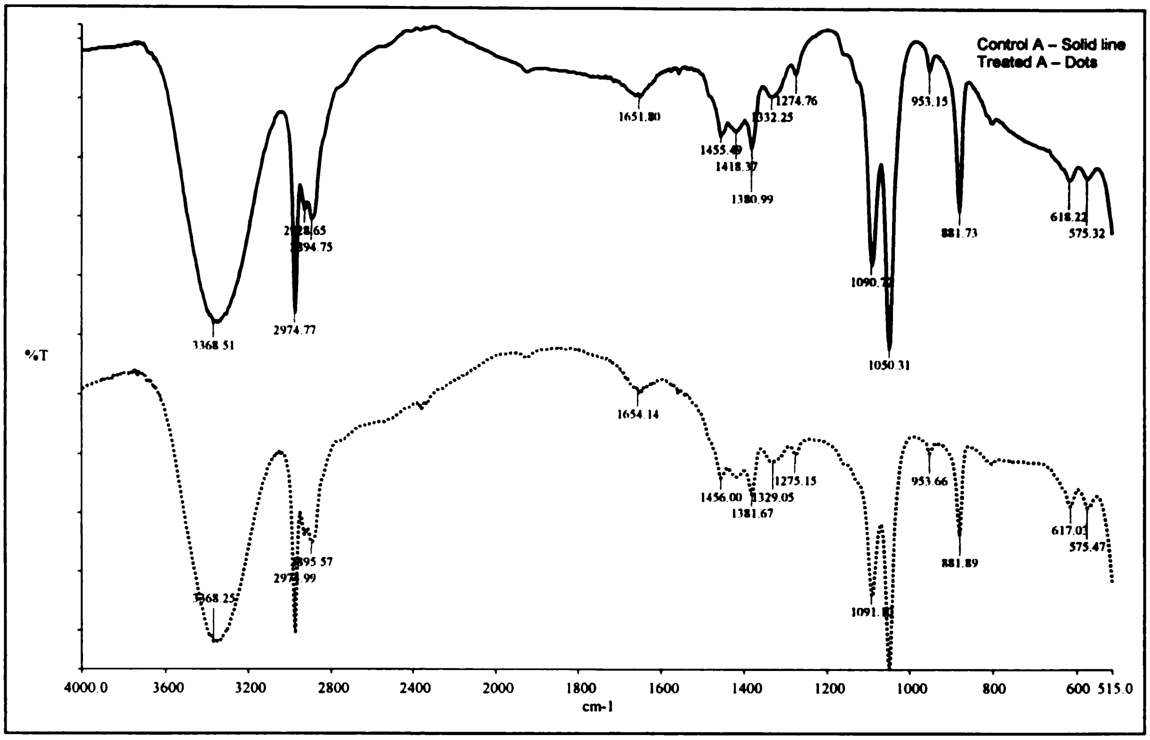


Figure D.1.7: Percent Transmittance of Substrate A in 95% Ethanol for 120 hours

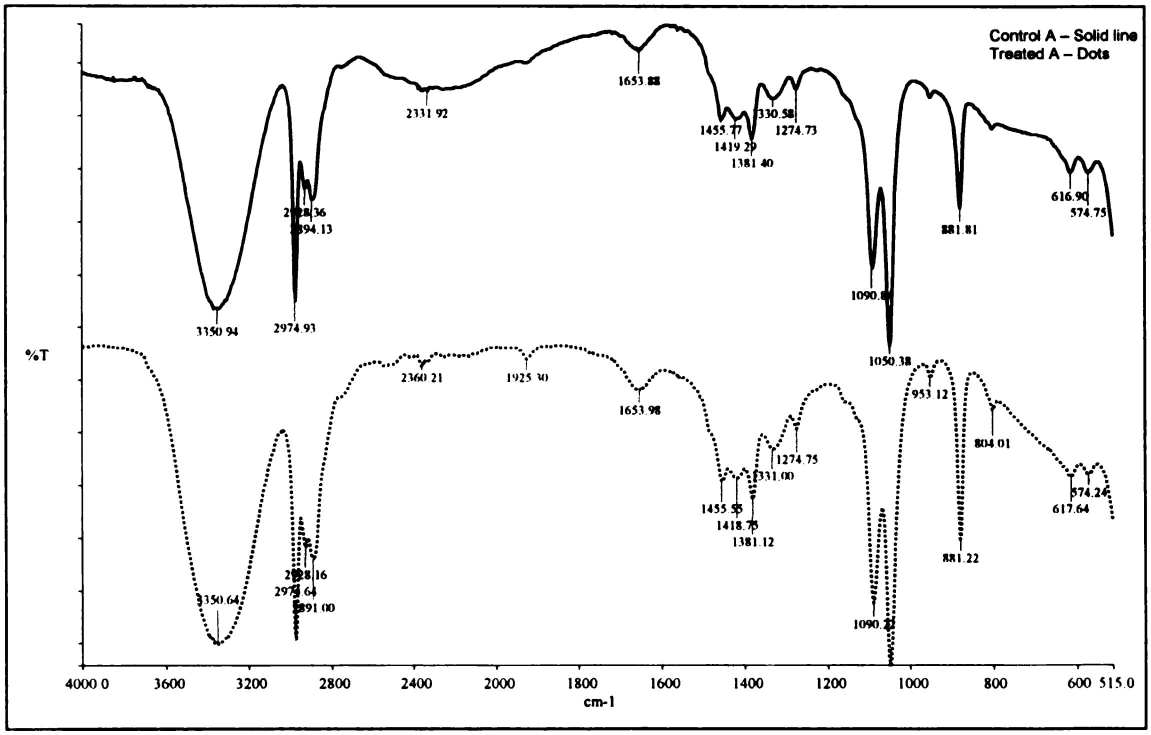


Figure D.1.8: Percent Transmittance of Substrate A in 95% Ethanol for 240 hours

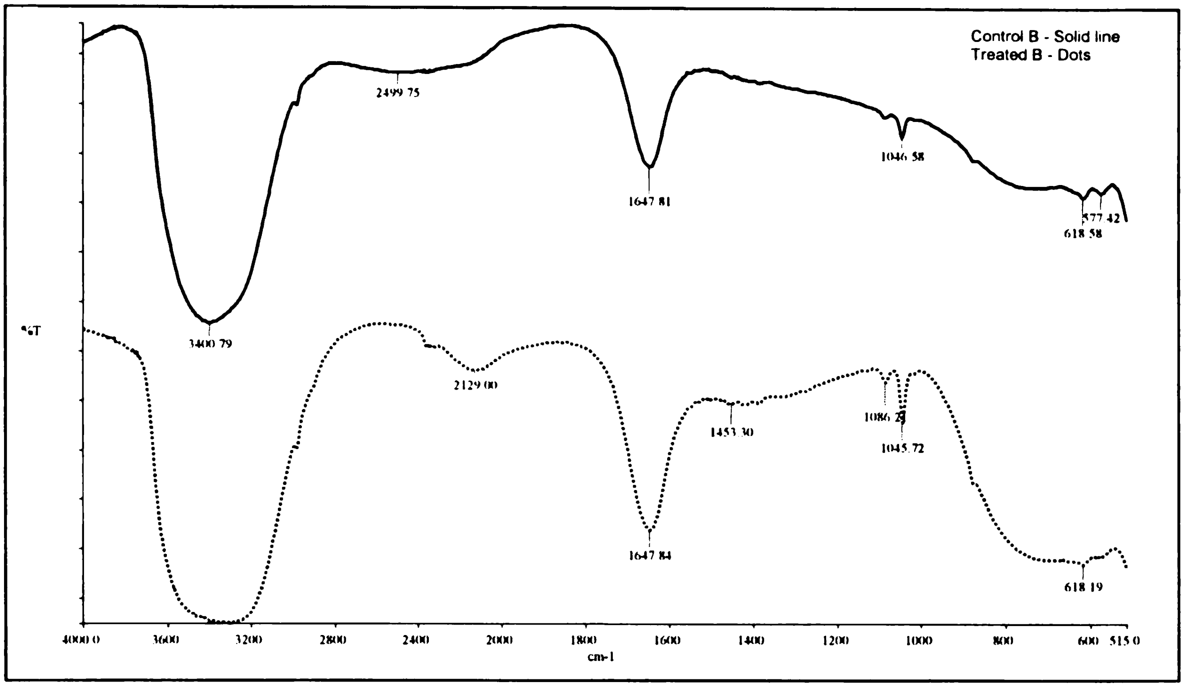


Figure D.2.1: Percent Transmittance of Substrate B in 10% Ethanol for 24 hours

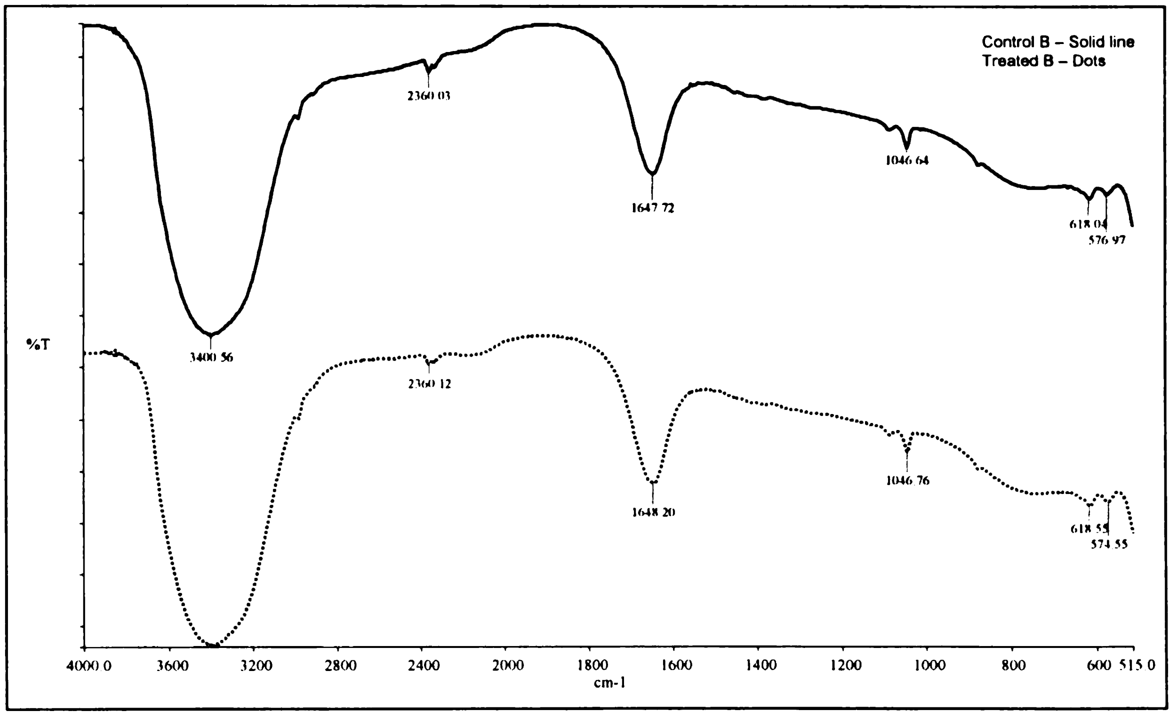


Figure D.2.2: Percent Transmittance of Substrate B in 10% Ethanol for 48 hours

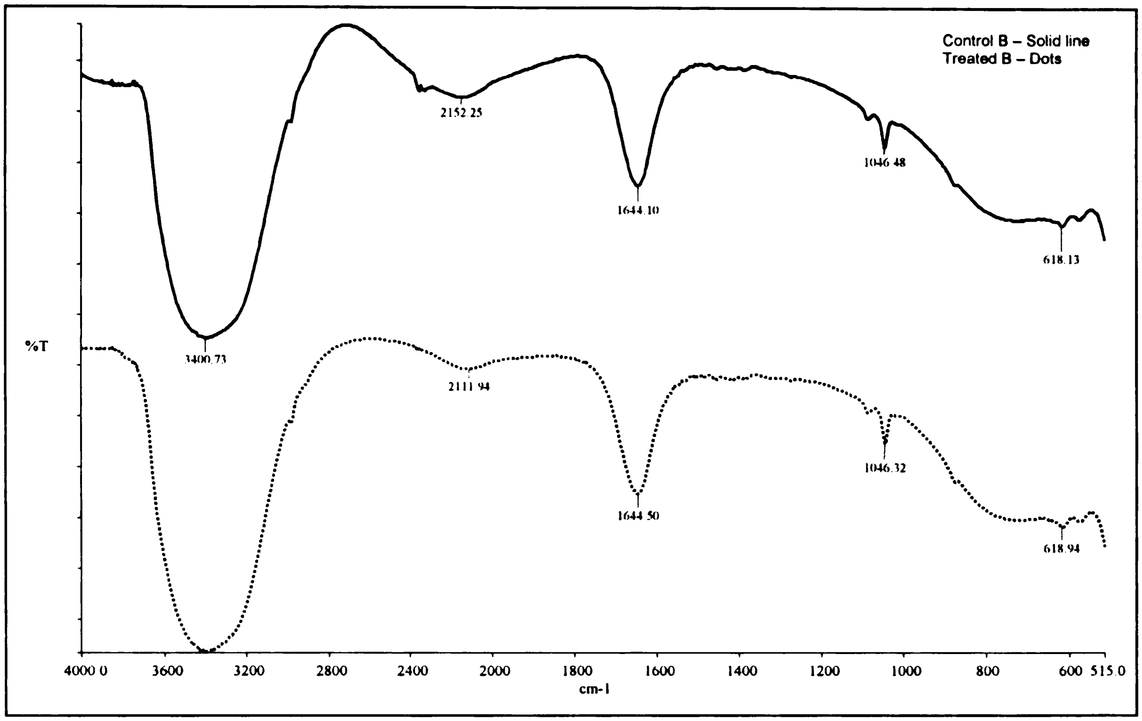


Figure D.2.3 : Percent Transmittance of Substrate B in 10% Ethanol for 120 hours

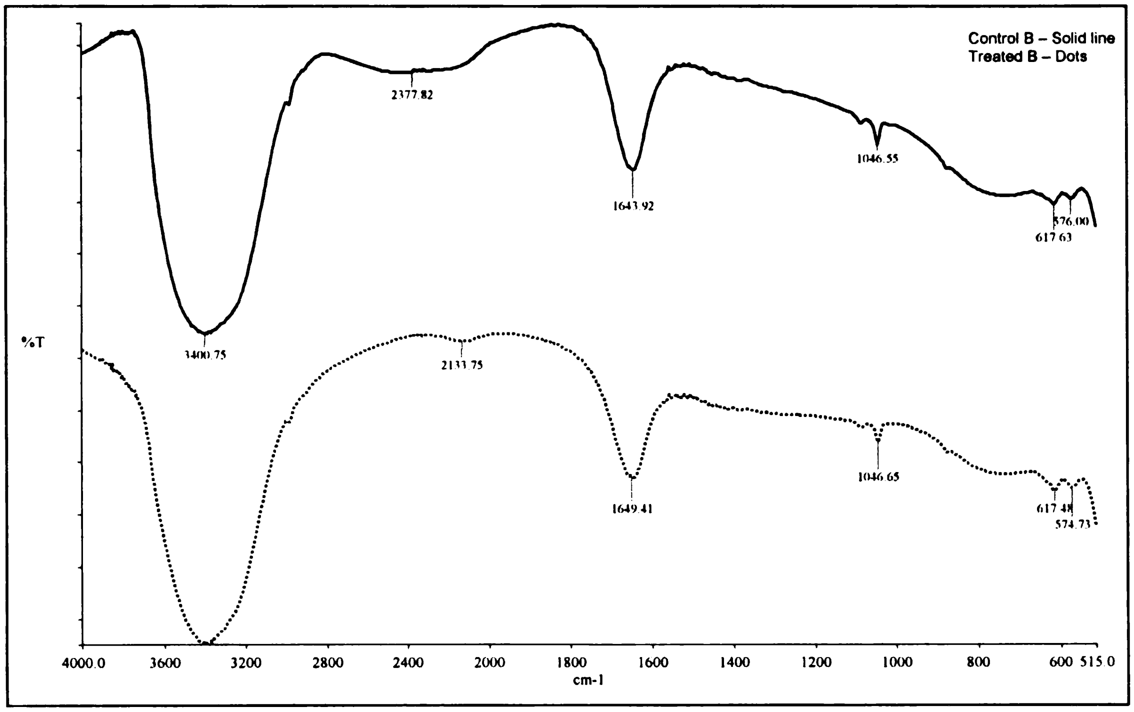


Figure D.2.4 : Percent Transmittance of Substrate B in 10% Ethanol for 240 hours

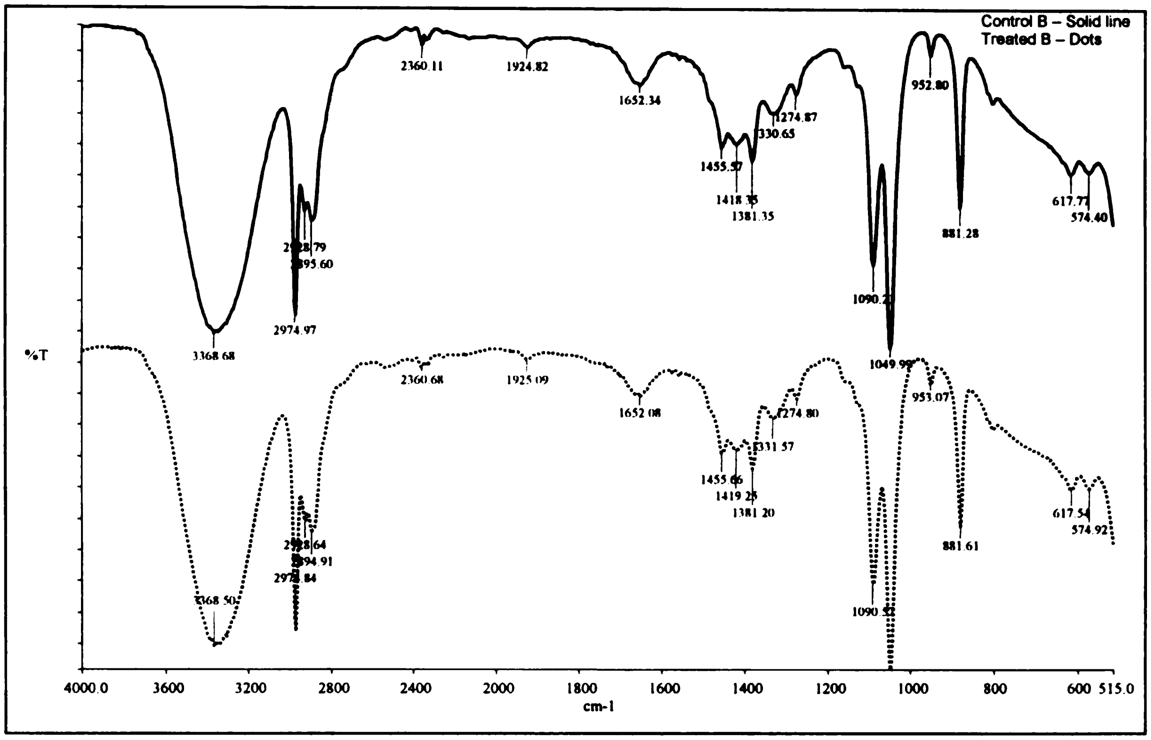


Figure D.2.5: Percent Transmittance of Substrate B in 95% Ethanol for 24 hours

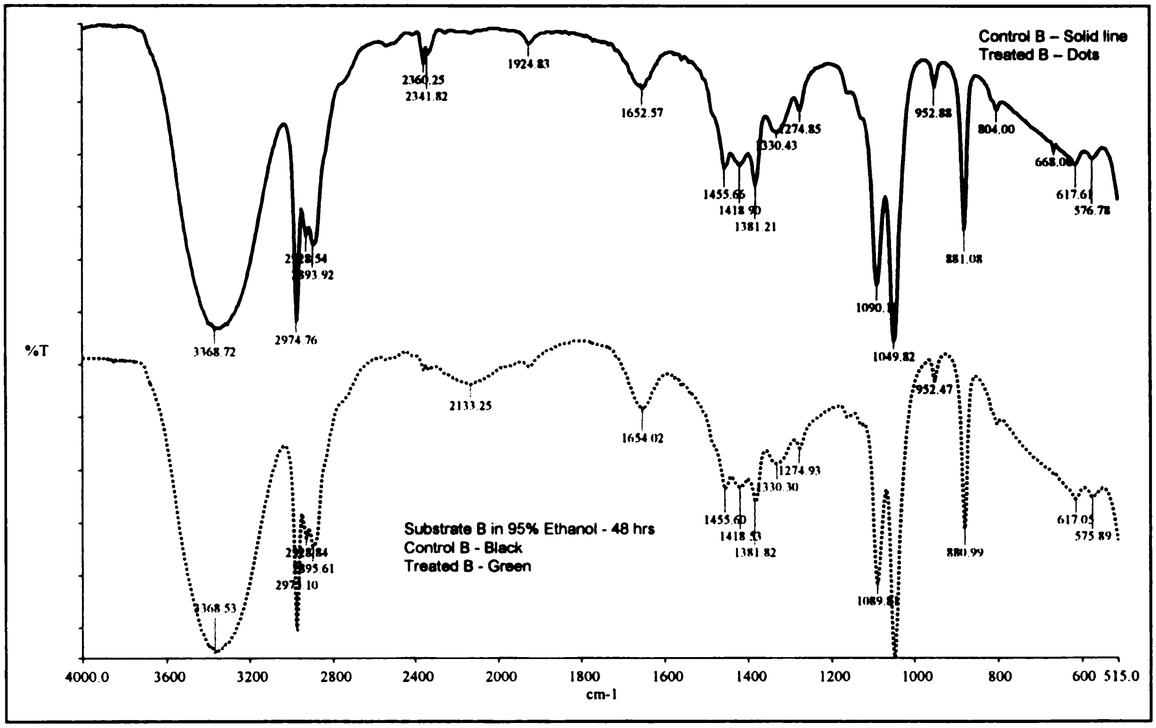


Figure D.2.6: Percent Transmittance of Substrate B in 95% Ethanol for 48 hours

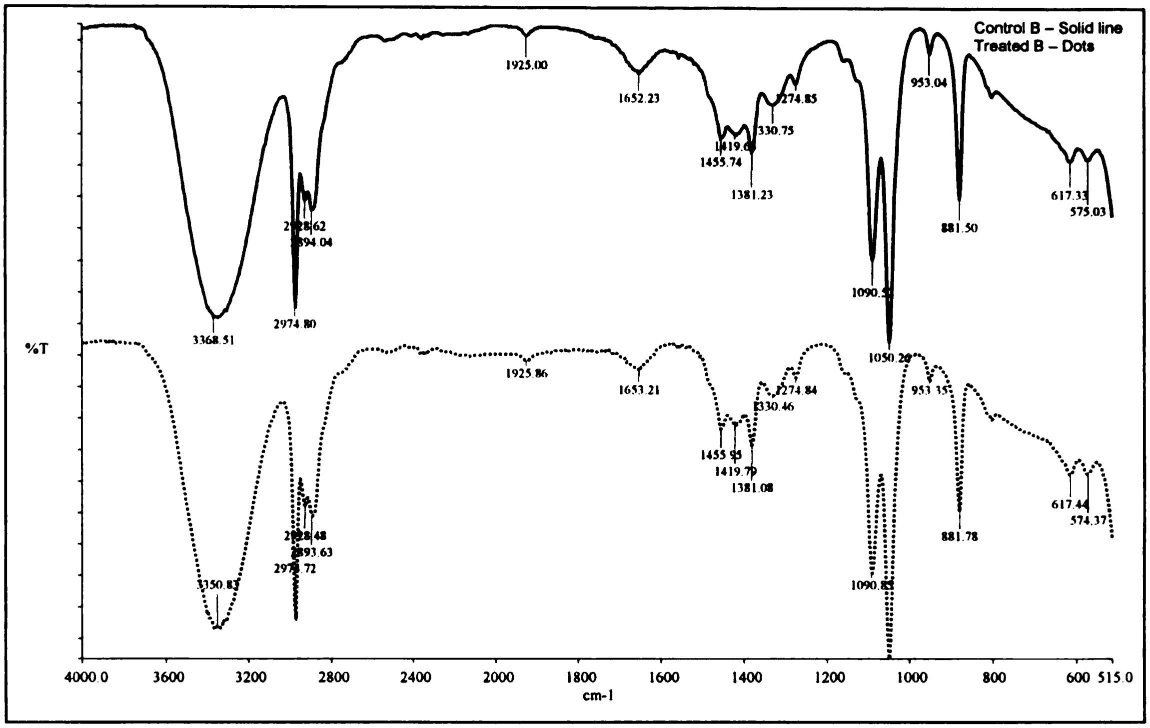


Figure D.2.7: Percent Transmittance of Substrate B in 95% Ethanol for 120 hours

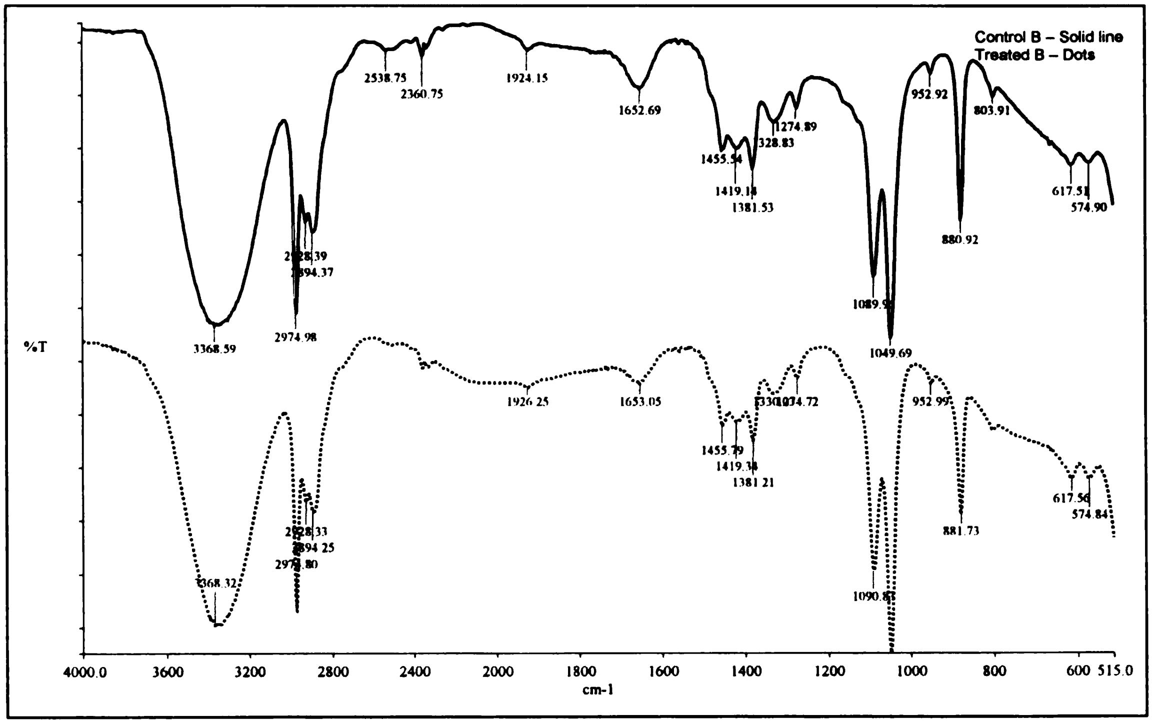


Figure D.2.8: Percent Transmittance of Substrate B in 95% Ethanol for 240 hours.

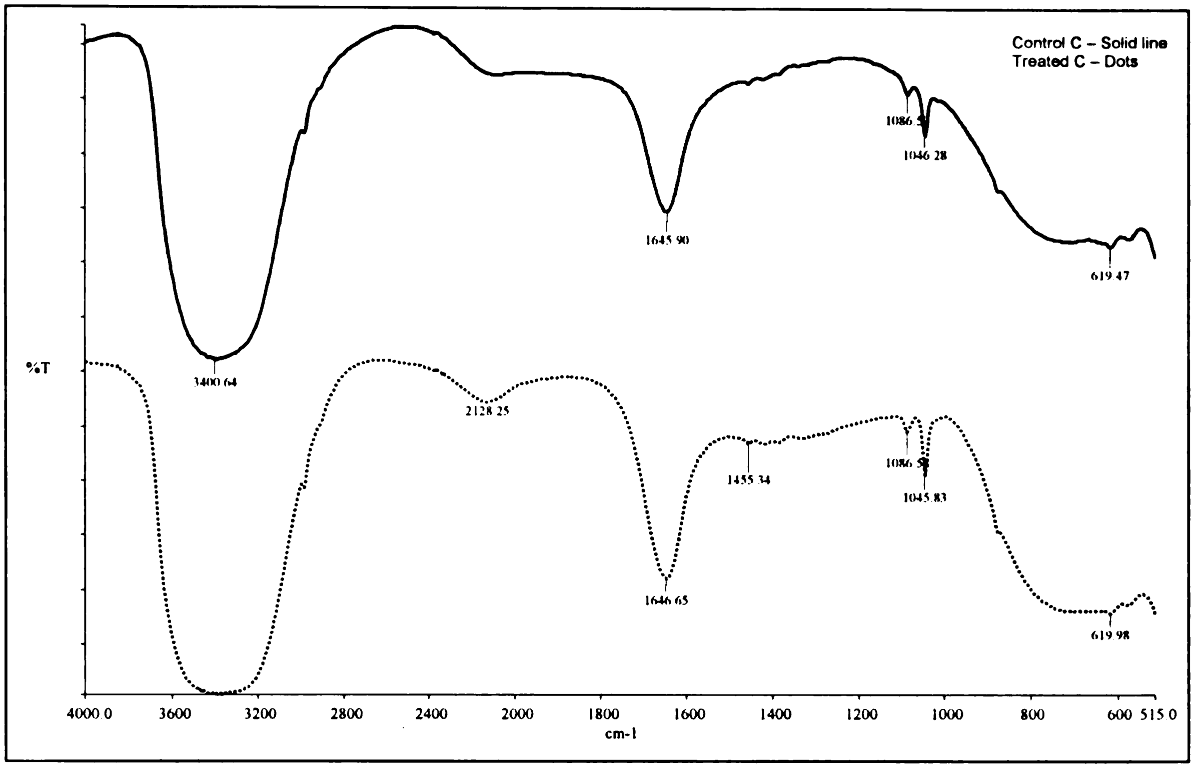


Figure D.3.1: Percent Transmittance of Substrate C in 10% Ethanol for 24 hours

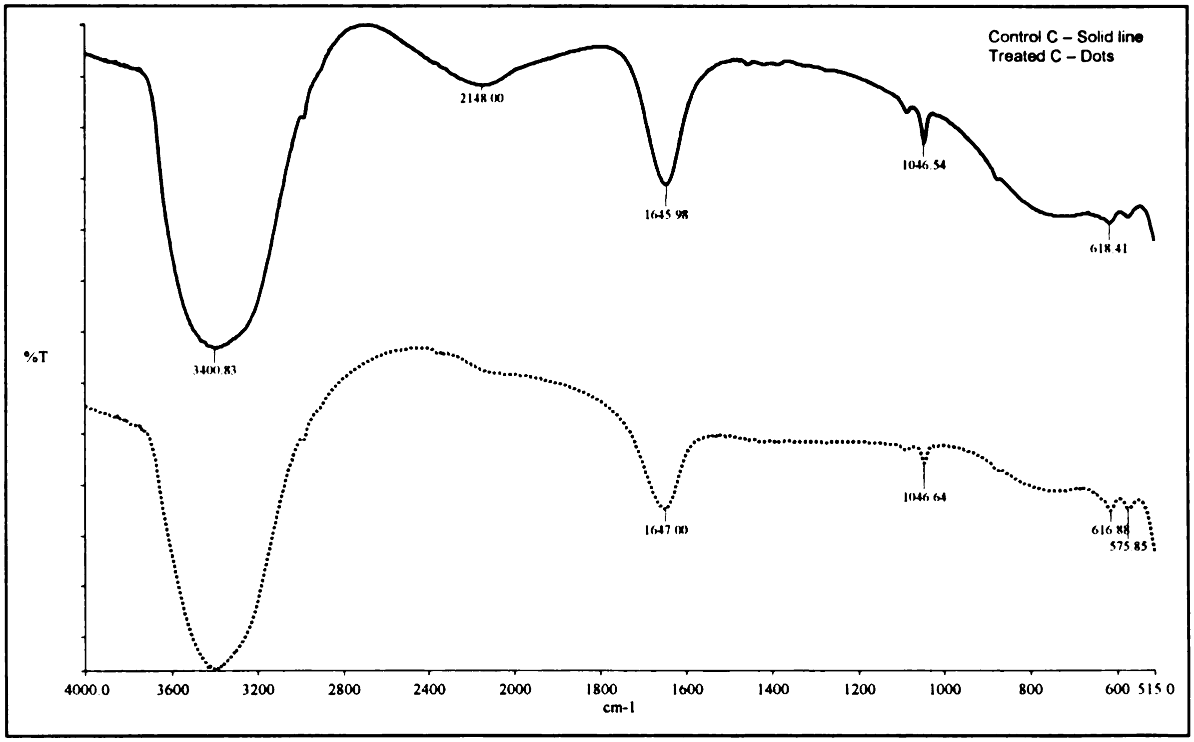


Figure D.3.2: Percent Transmittance of Substrate C in 10% Ethanol for 48 hours

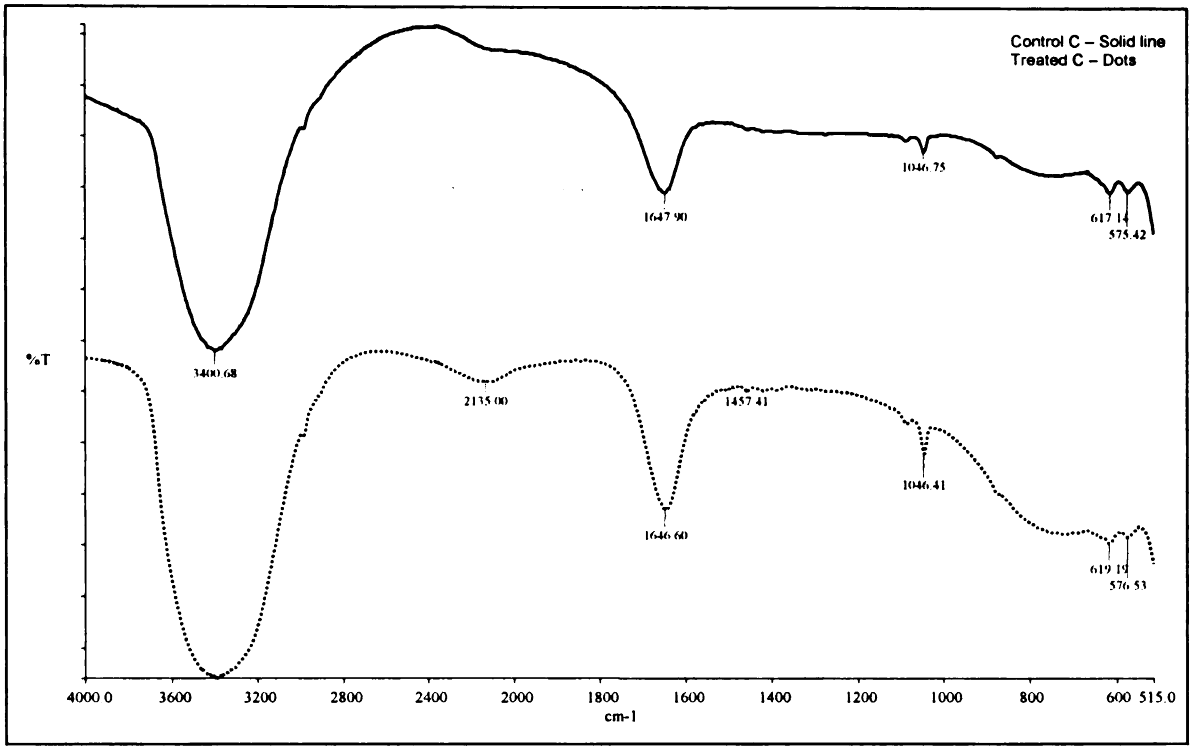


Figure D.3.3: Percent Transmittance of Substrate C in 10% Ethanol for 120 hours

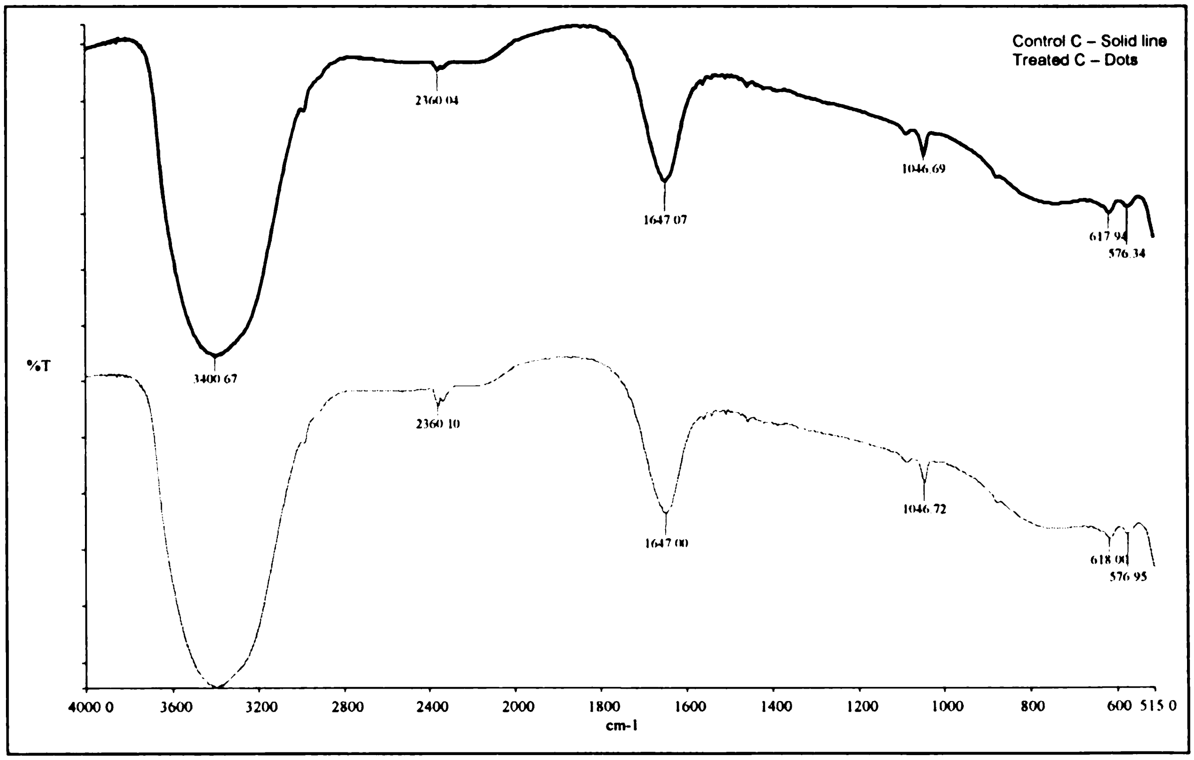


Figure D.3.4: Percent Transmittance of Substrate C in 10% Ethanol for 240 hours

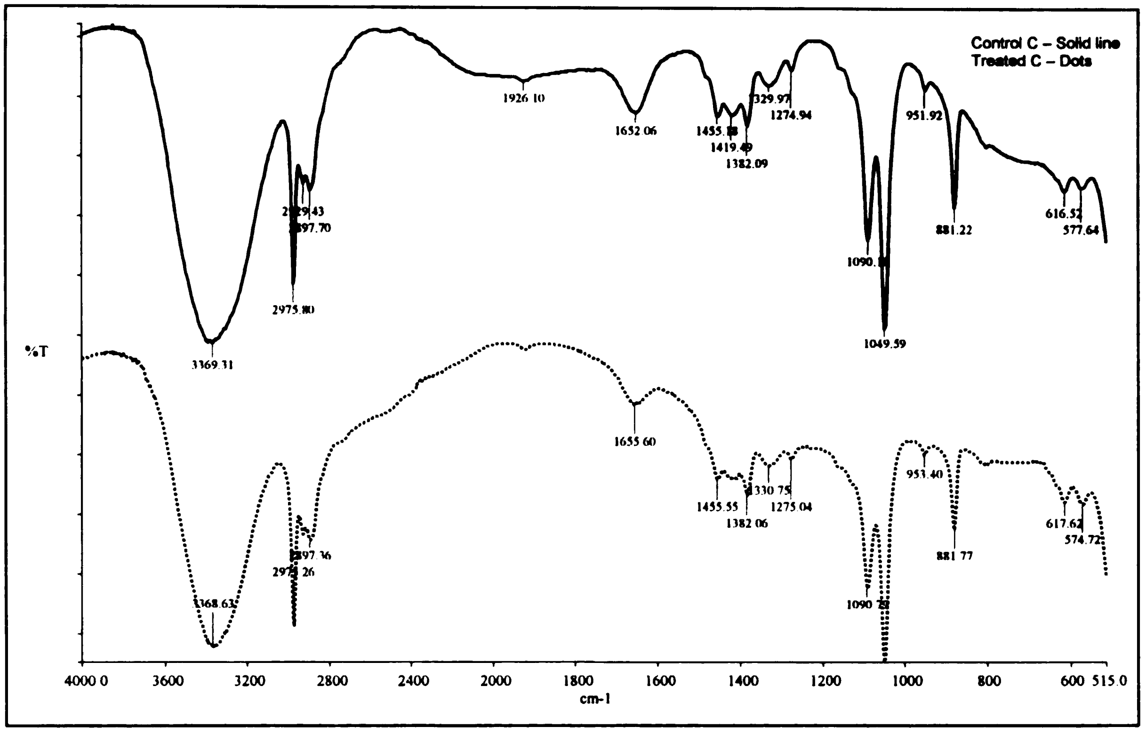


Figure D.3.5: Percent Transmittance of Substrate C in 95% Ethanol for 24 hours

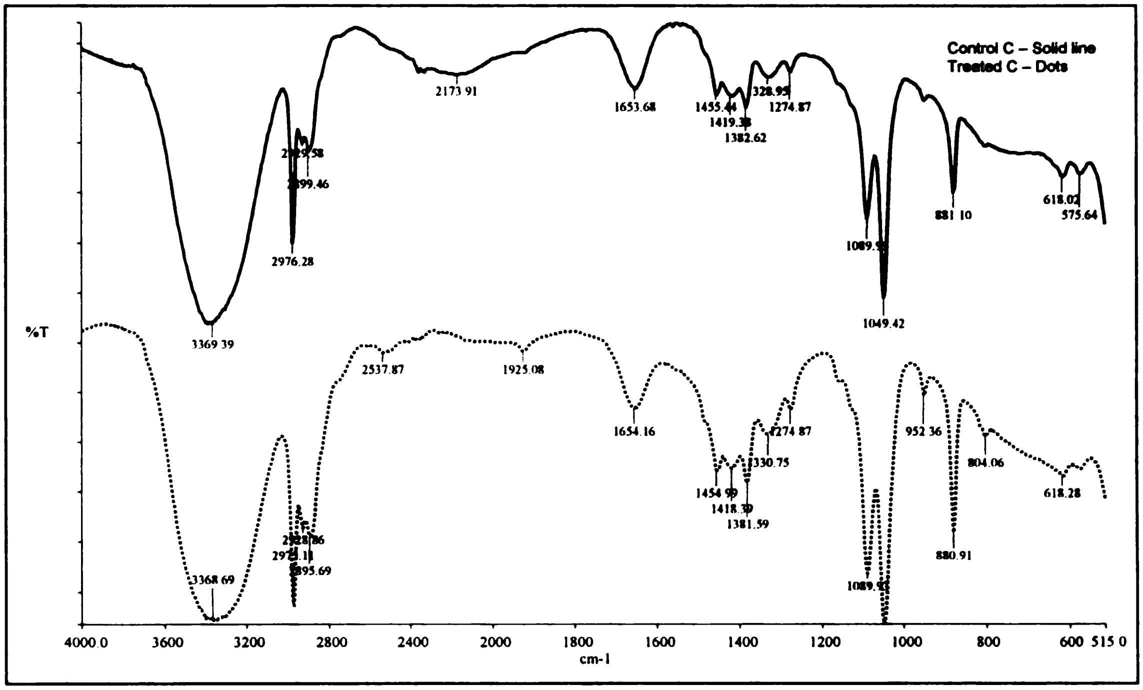


Figure D.3.6: Percent Transmittance of Substrate C in 95% Ethanol for 48 hours

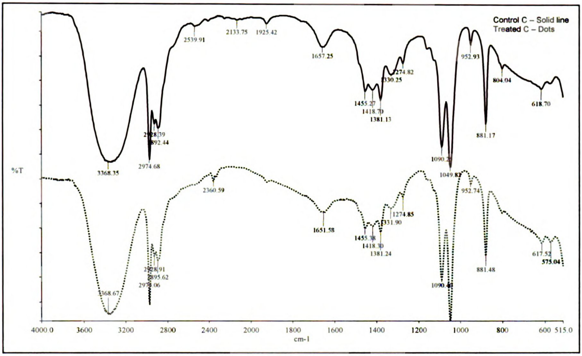


Figure D.3.7: Percent Transmittance of Substrate C in 95% Ethanol for 120 hours

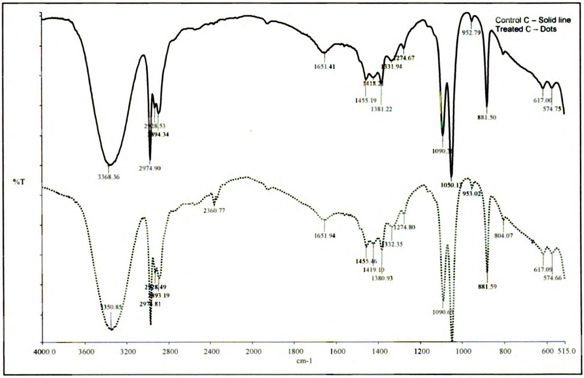


Figure D.3.8: Percent Transmittance of Substrate C in 95% Ethanol for 240 hours

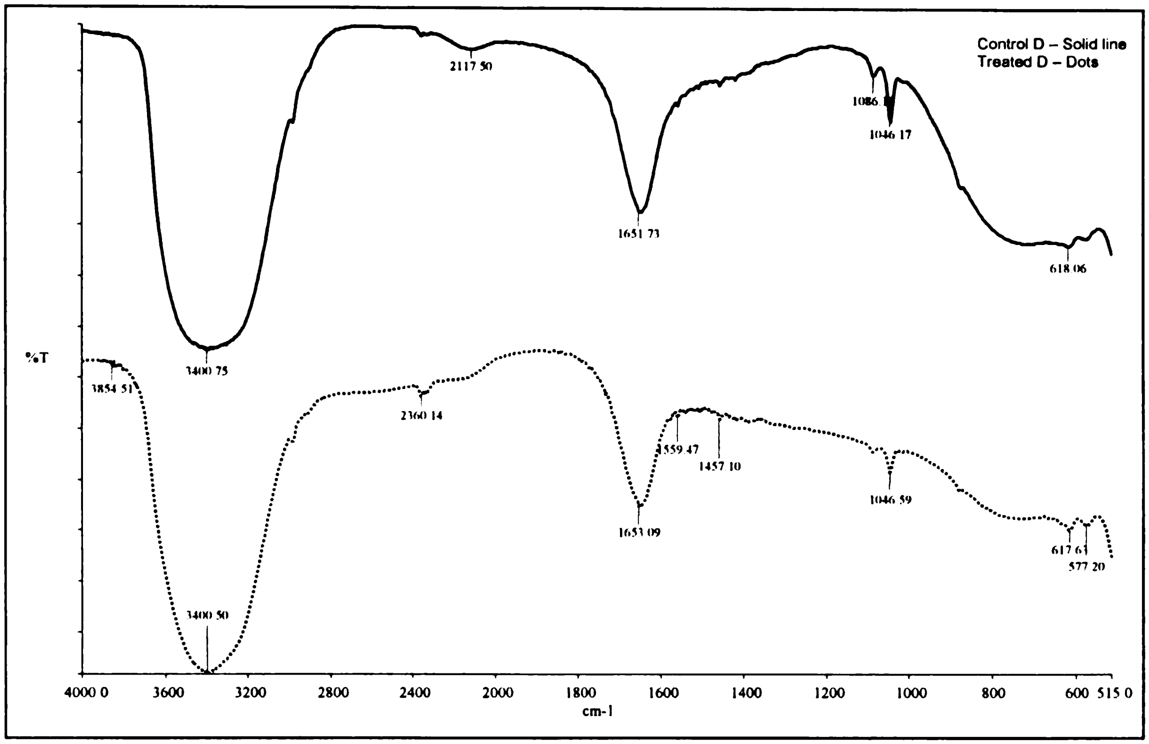


Figure D.4.1: Percent Transmittance of Substrate D in 10% Ethanol for 24 hours

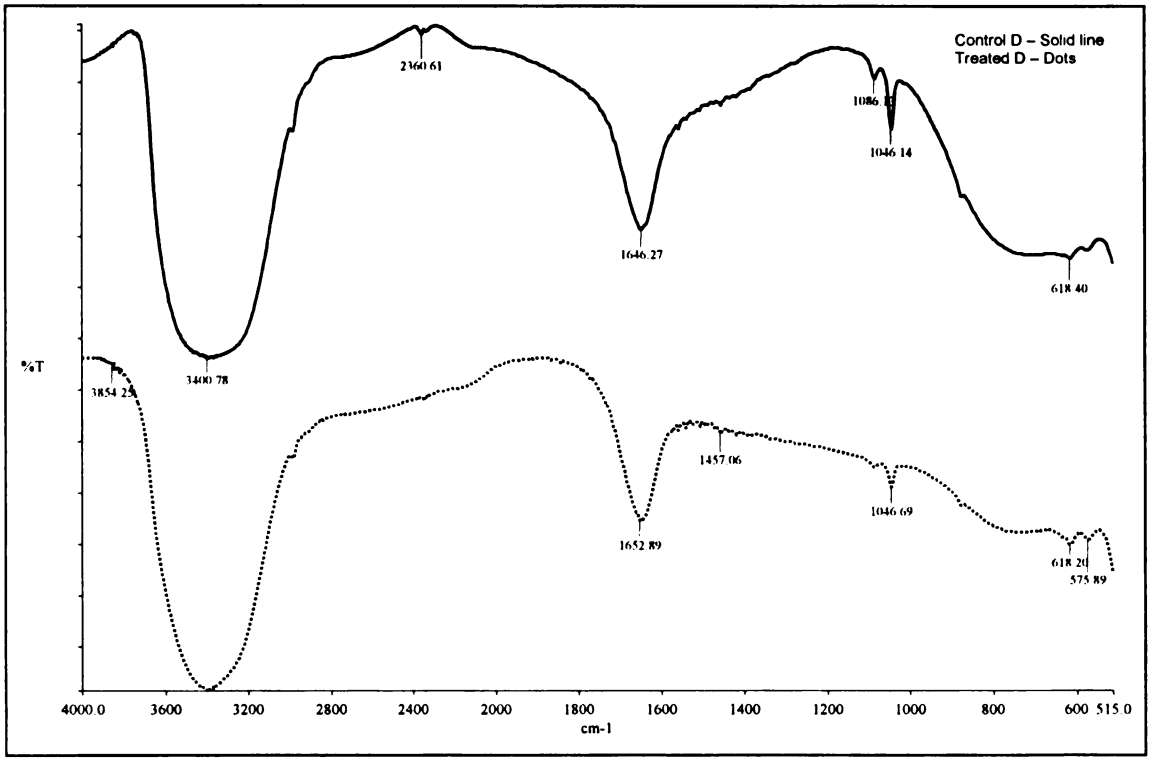


Figure D.4.2: Percent Transmittance of Substrate D in 10% Ethanol for 48 hours

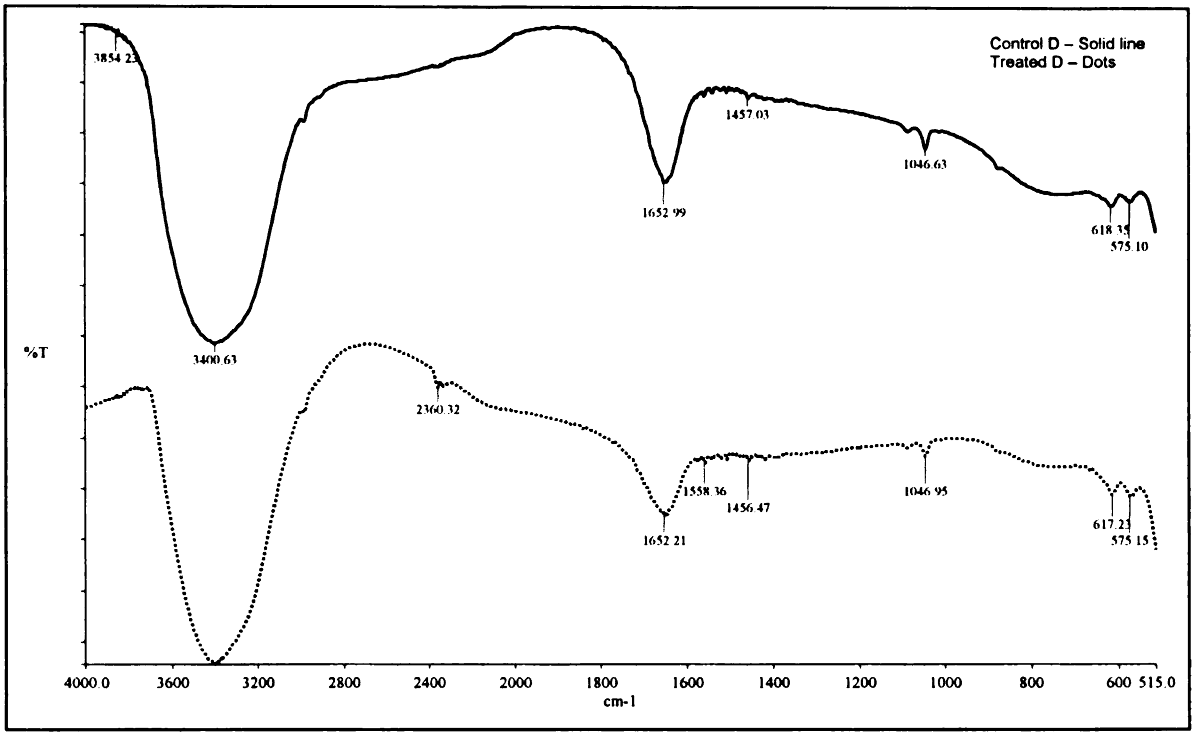
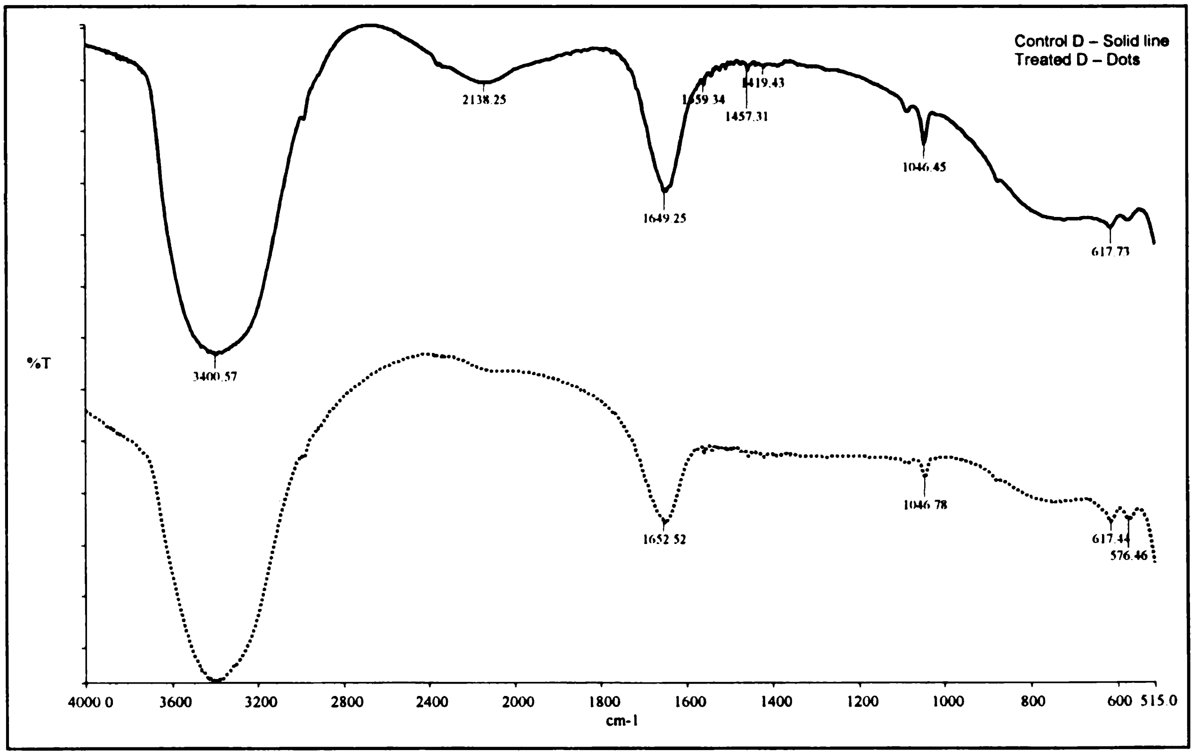


Figure D.4.3: Percent Transmittance of Substrate D in 10% Ethanol for 120 hours



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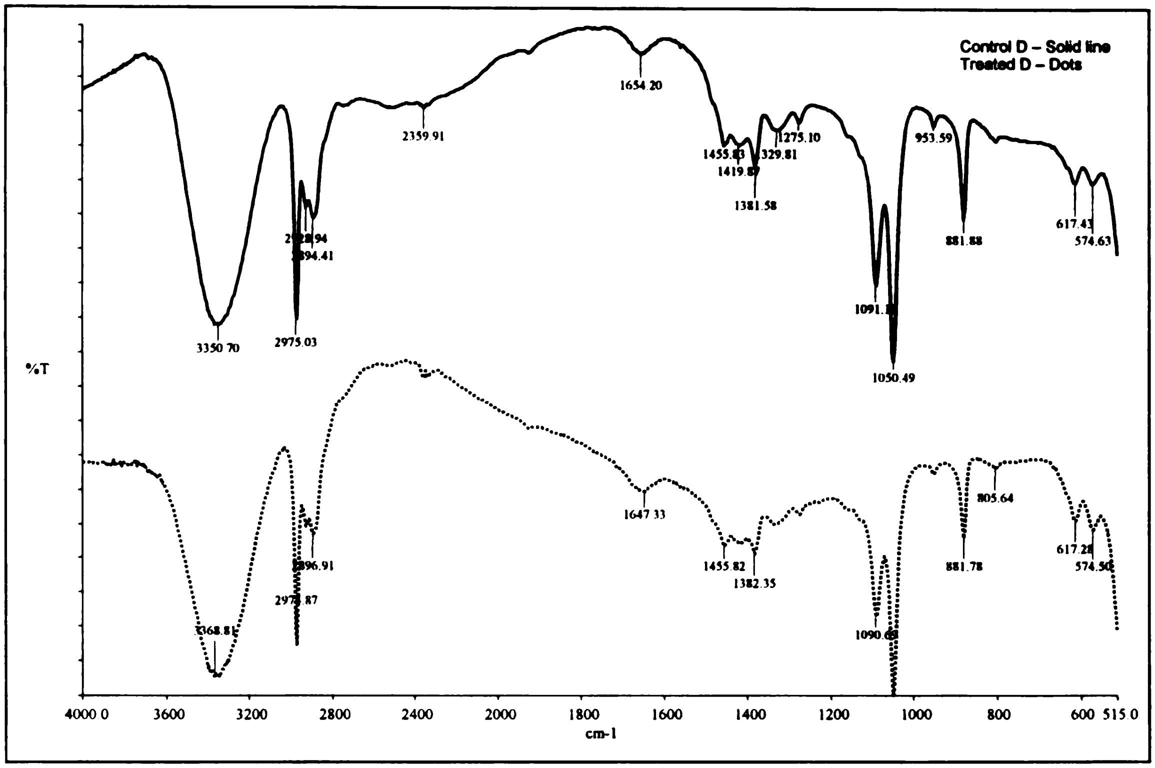

Figure D.4.5: Percent Transmittance of Substrate D in 95% Ethanol for 24 hours

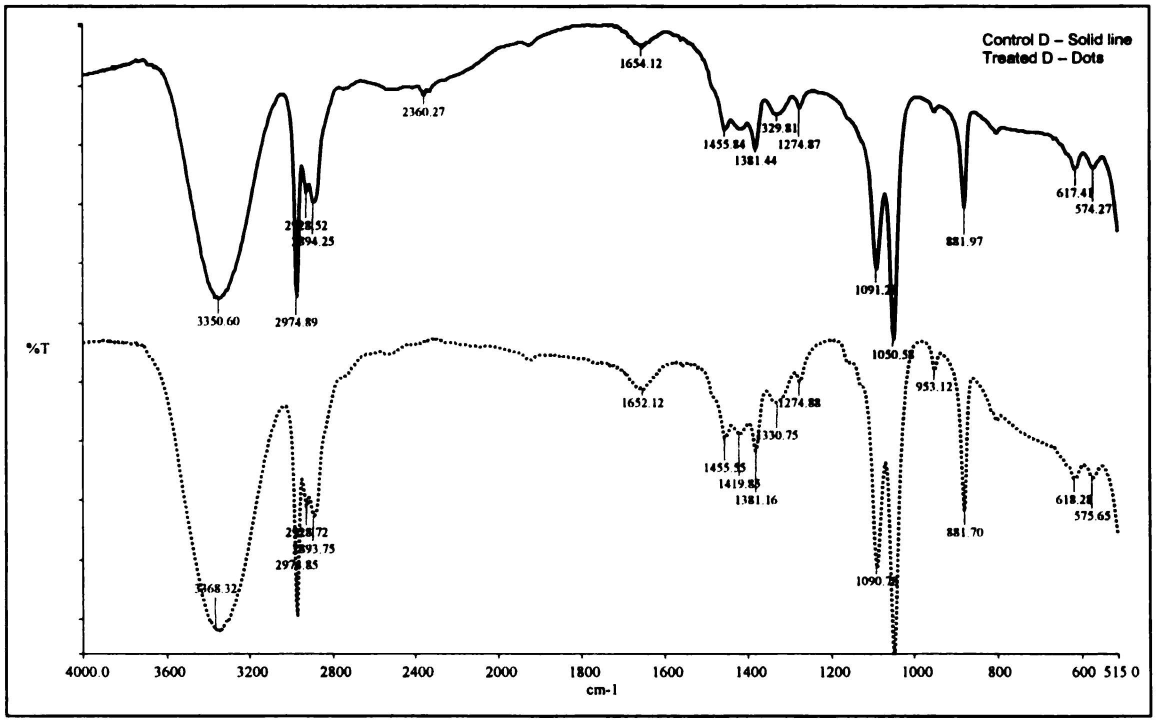

Figure D.4.6: Percent Transmittance of Substrate D in 95% Ethanol for 48 hours

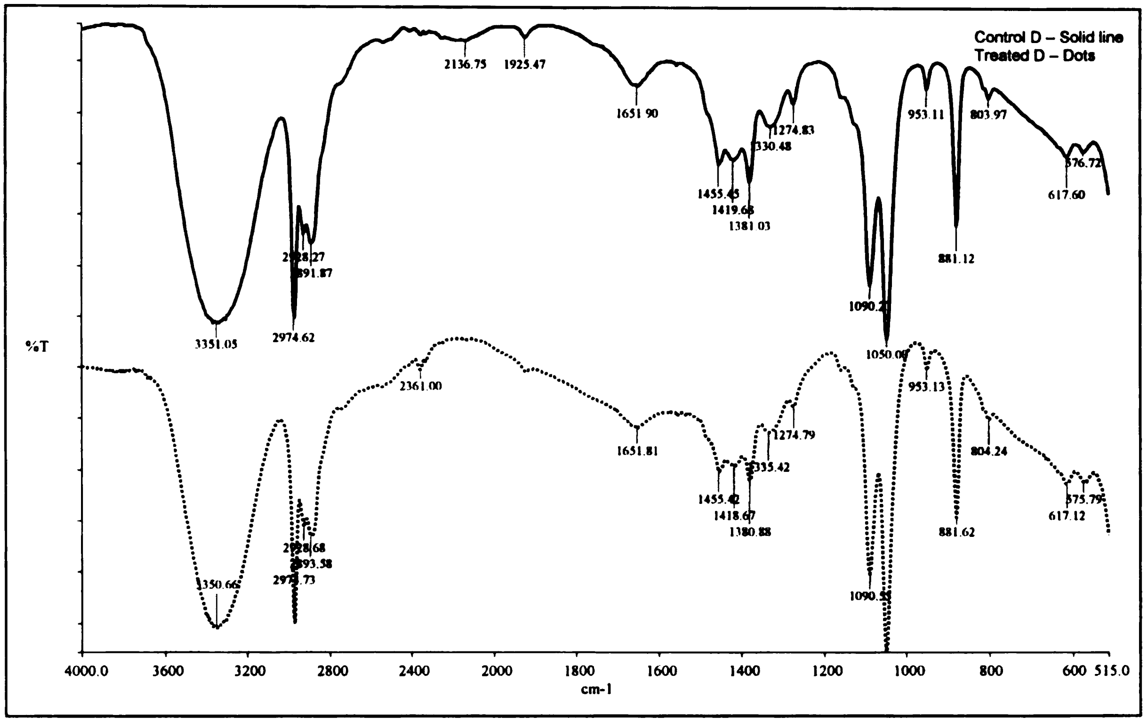

Figure D.4.7: Percent Transmittance of Substrate D in 95% Ethanol for 120 hours

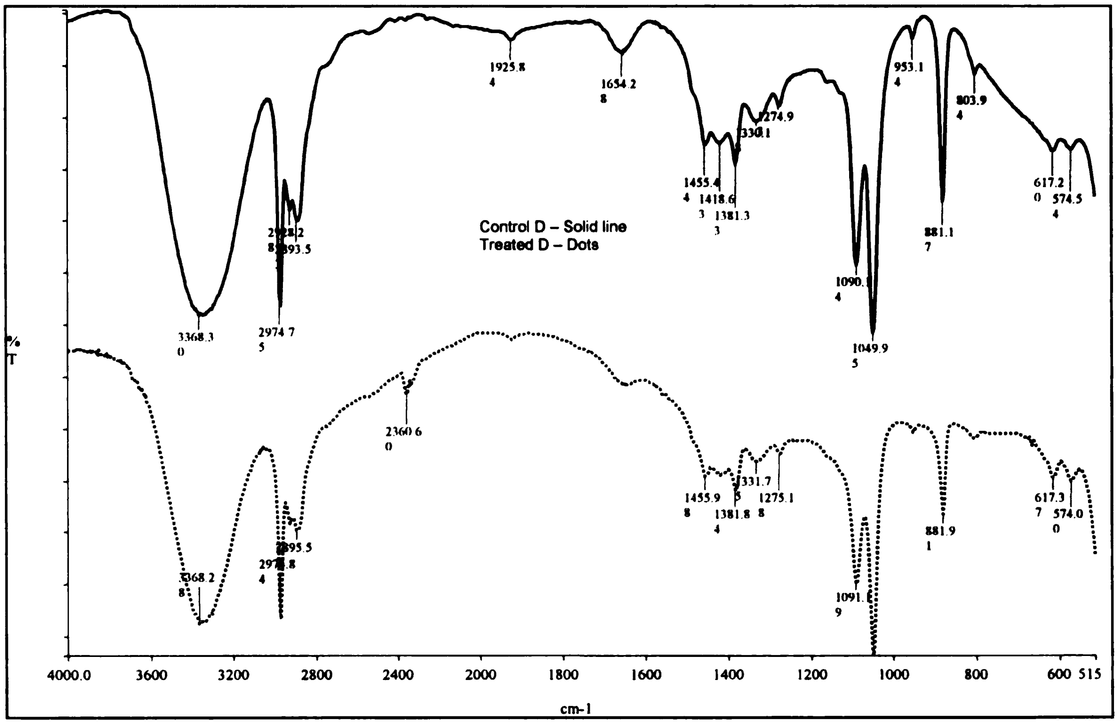

Figure D.4.8: Percent Transmittance of Substrate D in 95% Ethanol for 240 hours.

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