STUDY ON WOOD COATING

A major project submitted in the partial fulfillment of the requirement for the award of the Degree of

> Master of Engineering In Polymer Technology

> In the Faculty of Technology, University of Delhi, Delhi



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CERTIFICATE

This is to certify that Mr. Ashwani Kumar Neelam has satisfactorily completed the project work entitled "**Study on Wood Coating**" in partial fulfillment of the requirement for the award of the Degree of Master of Engineering in Polymer Technology of Delhi College of Engineering, Delhi during the academic session 2004-2006. This work has not been submitted in part of full in any other university or institution for the award of any other degree or diploma.

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ABSTRACT

This thesis deals with the preparation of solvent free wood coating composition cured with the EB radiation and the evaluation of its properties.

The known methods of coating comprise of coating with organic emulsions along with various solvents. Also the available UV radiation curing was not found suitable as the photo emission generates heat in the curing layer and gives rise to residual stresses.

It was found that the use of EB curing using free radical mechanism provide ultra fast curing even there is a bulk of the surface to be cured in comparatively less time than the other radiation processes available. Coating composition based on epoxy acrylate, cured at 400 KGy. The molecules of the coating get cross linked and provide protective surface to the wood.

The resulting DCE coatings hold better properties of gloss, stain (except acetone) and thermal properties in comparison to conventional coating. Also the increase in the mechanical strength of the coating and improved adhesive properties was noticed.

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ABBREVIATIONS

EBC	Electron beam curing
UV	Ultraviolet
VOC	Volatile organic compound
HAP	Hazardous air pollutants
Tk	Teak wood
Kl	Kail wood
Ki	Kikar wood
IR	Infrared
HDDA	Hexanediol diacrylate
DPGDA	Dipropylene glycol diacrylate
TPGDA	Tripropylene glycol diacrylate
PU	Polyurethane

Chapter 1

Wood is an important natural resource which finds applications in many areas like timber, furniture, pulp and paper industry etc. Wood comprises of cellulose fibers cemented together by lignin.

Despite many application of wood, it has certain limitations such as:

- Poor surface stability
- ➢ Inflammability
- Limited shelf life
- Poor abrasion resistance
- > Prone to attack by: moisture, UV radiation, microorganisms

These drawbacks of wood can be overcome by using specialized coating on the surface of wood or by impregnation of wood with monomer followed by polymerization. Presently, there are many coatings which are used for wood like nitrocellulose, polyurethane, melamine, etc. but these coatings have certain drawbacks such as:

- Thermal curing limits the range of coating, which can be used due to upper temperature limitation imposed by timber
- > Thermal curing is a time consuming process
- Solvent emission during curing pollutes the environment

Thus, radiation energy such as lasers generate a monochromatic energy source, focused infrared, UV sources or electron beam radiation may be used to create sufficient energy at the surface of the wood to produce drying and curing effect for the coating without affecting the underlying wood. Advantage of radiation curing over thermal curing is as follows:

- High Production rate
- Environment friendly technique
- Safe, reliable & energy efficient process
- > Does not require high temperature or pressure
- ➢ No solvent emission
- Cost effective

The present study aims at developing an electron beam curable coating composition suitable for wood, which will improve its properties. The benefits of EB curing are given as below:

- Energy delivered directly to molecules
- ➢ No residue or by- product formed
- Ambient temperature curing
- Cross-linking, grafting or polymerization is complete immediately during processing
- > Process is highly reproducible, controllable and precise

Radiation curable coating composition consists of

- Oligomers: Low or medium molecular weight resins, they provide the bulk of desired properties of coatings
- Reactive diluents: Mono or multi functional monomers, they reduce the viscosity of liquid film and contribute to the cross linking required in the cured film
- > Additives: They are used to impart some specific property to the film

Study was carried out on composition based on oligomers; namely, HDDA, DPGDA, TPGDA, etc. The effect of various reactive diluents and their concentration on the property of coated wood was studied.

2.1 BACKGROUND

Use of radiation energy curable coatings dates back to at least 4,000 years. Ancient Egyptians used a type of UV coating that cured when exposed to sunlight in prepearing mummies. Also, an asphalt-based oil coating that polymerized upon exposure to solar radiation was used by ancient Egyptians as a sealant for ships.

In the modern era, scientific interest in developing UV/EB-cured systems began in the 1940's. At that time, the first patent was granted for an unsaturated polyester styrene printing ink that polymerized under UV exposure. One of the first attempts at applying UV/EB-cured systems to manufacturing was made in the late 1960's, but successful commercial application did not evolve until the early 1970's. The publicized driving forces behind development of commercially viable systems were the energy crisis of 1974 and growing environmental concerns about VOC emissions resulting from conventional thermal-cure systems. However, the primary motivations for use of UV/EBcured systems were and still are improved product performance and increased productivity. Early applications of UV/EB-cured systems were limited to flat sheets, mainly in the wood products. Radiation energy cured processes of that time were limited by the need for a line-of-sight energy source. Starting in 1974, UV-curable inks and varnishes were used for decorating aluminum beverage cans. Improvements in plant engineering, such as rotating conveyors, multiple UV sources and adjustments to curing equipment, have allowed three-dimensional (3D) applications of UV/EB-cured materials. In addition, advances in polymer science have provided a wide variety of UV/EB-cured materials that can exhibit characteristics required by the end-user in specific applications.

2.2 WOOD

Wood is an important part of today's day to day life. It has wide areas of application ranging from structural timbers to small wooden artifacts. With rapid growth of population the demand of high quality wood is increasing and its availability is becoming scarce.

There are many limitations associated with poor surface stability, high inflammability, prone to attack by microorganisms etc. These limitations need to be overcome to improve the self life of wood based products. Thus, there is a need to upgrade low grade wood by application of specialized coating so that low quality wood can replace high quality.

Wood is a complex polymeric structure consisting of carbohydrates (cellulose and hemicellulose) which from the visible lignocellulosic structure of wood. Two kinds of wood are known as soft wood and hard wood. Soft wood contains a higher percentage of lignin (26-32 %) and a lower percentage of hemicellulose (14-17 %) than the hardwood, which contain 17-16 % lignin and 18-27 % hemicellulose. There are other ways by which woods differ from one another like colour, grain/pore structure, nature and content of constituents, density and growth characteristics etc.

2.3 WOOD COATING

The term wood coating refers to the system applied as single or multiple layers to substrates with aesthetic and protective functions. There are several kinds of wood coatings like nitrocellulose, PU, melamine, polyester acrylates, urethane acrylates, epoxy acrylates etc in use today. The techniques used for wood coating are mainly based on drying and curing of lacquers using air circulating ovens. This restricts the technique for coating that can be used for heat sensitive substrates like timber. Also, conventional coatings available today are mainly based on lacquers dissolved in organic solvents. The curing of such coatings is time consuming process and also emits a lot of volatile matter .Thus, for a system there is a need of coatings, which are solvents free and less time consuming. The use of radiation energy may provide a solution to these requirements. Radiation energy may create sufficient energy at the surface to create drying and curing effects for the coating without affecting the underlying timber. Both ultraviolet (UV)

energy and electron beam (EB) energy can be used forms of radiant energy and can be used for above purpose.

2.4 CONVENTIONAL COATING

Conventional oil/solvent based and waterborne coatings are evaporative systems. They use suitable partially pre-polymerized monomers (alkyd, acrylic, epoxy, silicone, etc.) and pigments dispersed in a solvent. These pre-polymers must further polymerize and cross-link during the curing process to become solids and the solvent system gets evaporated.

Conventional pre-polymers are formed by a chemical condensation reaction at elevated temperature. This reaction occurs at a chemical plant or coating, manufacturing facility, and not during the cure. Just before the pre-polymer gets long enough to form a gel, the temperature is reduced and the pre-polymer is dispersed in an organic solvent. The organic solvent is usually VOC and often accounts for more than half of the volume of liquid coating. Water accounts for a similar fraction of the volume of water based coatings. Some conventional coatings even contain HAP.

When conventional materials are applied and the organic solvent, pre polymerized resin molecules are able to contact each other, solidification of the coating occurs as the prepolymer further proceeds in its polymerization by a chemical reaction. This is a relatively slow process because the temperature is now much lower than it was in pre polymerization and, in fact, it is a process that never ends.

Conventional materials use infrared (IR) radiant energy (i.e., heat) to cure. It is electromagnetic radiation with a wavelength between 1000 nm and 11,000 nm. Visible light is electromagnetic radiation with wavelengths from 400 nm to 700 nm. Microwaves are electromagnetic radiation with wavelengths in meters (See Figure 1). IR energy is sensed as radiant heat and its wavelength is based upon temperature. Heat is used to evaporate organic solvent or water and to accelerate polymerization.

At ambient temperatures, evaporation and polymerization can take from hours to days. Thermal ovens can reduce evaporation and polymerization time to minutes. However, thermal ovens are bulky, take up considerable floor space, and consume large amounts of energy because they must heat the air flow through the oven, in addition to heating the coating, ink, or adhesive and substrate.

THE ELECTROMAGNETIC SPECTRUM

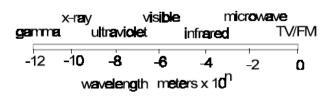


Figure 1

2.5 RADIATION CURING

Radiation cured coatings have replaced many conventional low solids, solvent borne coatings, Radiation curing uses electron beam (EB), ultraviolet (UV) light, or visible light to polymerize a reactive and usually solvent-free coating material.

Although the final properties of radiation cured coatings are often superior to other systems, the reason for their popular growth has been primarily due to improvements in productivity and no environmental emissions. However, radiation cured coatings are having a difficult time emerging from their early status as a niche product.

The primary applications for radiation cured polymers include inks, adhesives, and coatings where coatings are by far the largest segment. Some of the more important coating applications are found in every day products such as hardwood flooring, metal and wood furniture, electrical wire and cable, release papers, beverage cans, magazine covers, packaging, leather finishes and computer magnetic media

There are many benefits & limits of UV/EB curing

2.5.1 Benefits

- UV/EB technology allows to improve productivity. The curing on expose to UV or EB radiation is a lot faster than drying times, involved with conventional solvent or water-based coatings. So the product is immediately ready for testing and shipment. Moreover the rapid cure speed allows one continuous process instead of a multi-step process including intermediate drying
- 2. It is also an environment friendly technology without any VOC emissions and no flammability and the energy consumption of the process is lower
- 3. EB coatings can be used for heat-sensitive substrates (wood, etc)
- 4. Very fast production, cures in milliseconds

Even with the above mentioned advantages, radiation curing technology presents also certain limitations:

2.5.2 Limitations

- UV generates some heat (infrared range) during cure. This is a disadvantage to some heat sensitive parts but can be an advantage to coating systems that require additional thermal energy to cure
- 2. Some raw materials are skin irritants until they are fully cured
- 3. Complex geometries, thick or opaque coatings face problems in curing

2.6 CURING OF UV/EB MATERIAL

Electron beam curing mechanisms are similar to the UV-free radical mechanism. EBcured materials need EB radiant energy to polymerize monomers and oligomers into a solid. However, the electrons are accelerated to a much higher energy state, and the electron itself has sufficient energy to initiate polymerization. The impact of these electrons is high enough to break chemical bonds and to generate ions. The ions then transform themselves into free radicals, which then initiate polymerization. Thus, the EB mechanism requires no photo initiator. There is also greater penetration of the radiation (greater depth of cure) with less interference from pigmentation. UV-cured materials need UV radiant energy to polymerize monomers and oligomers into a solid. There are many polymerization mechanisms; however, currently only two polymerization mechanisms are used for curing UV/EB materials: free radical polymerization and cationic polymerization. A free radical cured UV material must be either very thin, clear, or both in order to let sufficient amounts of UV radiant energy to penetrate all the way through the material. A free radical cured EB material can be filled, pigmented and thicker because EB penetrates more than UV. Cationic photoinitiators, after UV/EB exposure, spontaneously form cations that trigger further cationic polymerization. Once the cationic polymerization has been started, cationic reactions can carry polymerization to completion in thicker, opaque materials or even in the dark (i.e. after radiant energy exposure stops).

UV/EB monomer and oligomer molecules have functional groups that attach to the side of a carbon chain. These are also referred to as pendant groups. Pendant groups aid in reducing the energy that must be added to polymerize and to form crosslinks. Crosslinks interconnect carbon chains and, as a result, provide greater mechanical strength, resistance to abrasion, higher softening temperatures, and increased chemical resistance.

Polymerization and crosslinking are the critical processes that take place as UV/EB material cures. Free radical curing is very fast and may reach completion in a millisecond or less. Cationic curing is slower and may take a few seconds to reach completion. Fortunately, cations are more stable than free radicals and endure much longer. Monomer and oligomer molecules are initially short enough to be mobile and, therefore, are in a liquid state. As polymerization progresses, polymer chains grow in length, become less mobile, and form a gel. In the gel state, a polymer solidifies, but has no significant mechanical strength. As polymerization continues, crosslinking occurs and the polymer gains mechanical strength. A material is fully cured when polymerization and crosslinking processes are virtually completed.

2.6.1 Free Radical Polymerization

In free radical polymerization, a monomer or oligomer joins with a free radical and, in effect, forms a larger free radical. This larger free radical then acts upon another

monomer or oligomer and forms an even larger molecule, and so on. The process is a chain reaction (another term used by chemists and physicists to mean a continuing reaction), that is endless until a polymer molecule is terminated. Termination occurs when,

- 1. One chain bonds endwise to another, which creates a pair of electrons that will not react further
- 2. An oxygen atom reacts with the free electron in a free radical and creates a pair of electrons that will not react further
- Polymer molecules limit the mobility of monomer molecules so that the reaction stops due to the unavailability of reactants, and the free radical reaches the end of its lifetime

The second termination mechanism is also referred to as oxygen inhibition. Note that this is only an issue in free radical polymerization, not cationic polymerization. Oxygen inhibition is avoided by blanketing the reaction area with dry nitrogen to exclude oxygen that would otherwise be present in ambient air. The presence of oxygen molecules could cause polymer chains to be terminated too short, or limit crosslinking. The types of monomers or oligomers define the structure of the polymer and, therefore, its properties. The basic properties are modified by crosslinking that molecularly bonds the polymer chains together while we might like to think that a time integrated dose of UV/EB radiant energy causes a cure, intensity (the rate at which photon energy is absorbed) is also important. Molecules at the surface (the topmost level) may receive 100 or more times the intensity of UV energy as molecules at the bottommost level adjacent to the substrate. As a result, adhesion failure may occur if bottommost coating molecules do not receive enough radiant energy to cure.

A free radical is a molecular fragment that has a single free valence electron

Photoinitiators are unsaturated molecules (having aromatic or aryl carbon rings) that form free radicals very readily. Valence electrons are unreactive when paired with another valence electron; however, a single valence electron always seeks a second valence electron. A free valence electron is formed by homolytic fission. This occurs when sufficient UV radiant energy is absorbed by the photoinitiator molecule. Homolytic fission means that both fragments are free radicals with single electrons available. Free radicals exist for a limited time and must find and react with monomers and oligomers within their lifetime. When a free radical captures an electron from a monomer or oligomer, that monomer or oligomer becomes part of the free radical. A single valence electron is always left free after this joining and this new expanded free radical reacts again and again as it forms a polymer. This is a chain reaction. In this way, monomers and oligomers form polymers that contain long chains of monomer molecules. Free radical polymerization contains the following steps:

Free radical polymerization contains the following steps:

Initiation R ———————————————————————————————————
Propagation $R^* + R_1 \longrightarrow RR^*_1$
Chain Transfer RR*, + AH> RR, H + A*
Termination $R_n^* + R_m^* \longrightarrow R_n R_m$
Initiation

Figure 2 Free Radical Polymerization

2.6.2 Cationic Polymerization

Cationic polymerization is an ionic process that is not inhibited by oxygen and, therefore, can cure in air without a nitrogen blanket. Cationic polymerization will continue after exposure to UV/EB radiant energy source ceases. Therefore, the only concerns are to initiate cationic polymerization and to store the product for a few seconds to allow for the cure to complete.

Cationic photoinitiators are unsaturated molecules that break apart by heterolytic fission under UV/EB radiant energy to form cations and anions. Heterolytic fission means that the fragments are different.

Protons or absorbed electrons are attracted to a cathode just as hydrogen ions are. The cations also cause fission to continue until all available photoinitiator molecules have ionized. Therefore, once started, polymerization will spread throughout the UV/EB material. Unlike free radical polymerization, cationic polymerization is an ionic reaction that cannot be terminated by a reaction with oxygen molecules. Because anions are also formed, the overall state of the polymer is neutral, even though polymerization occurs because cations are present. Because cationic photoinitiators now exist, the list of potential applications that can be served changes and expands. These cationic photoinitiators can be used to polymerize coatings that because of absorption, shadowing, or pigmentation, cannot receive enough radiant energy throughout their depth. Once cationic polymerization starts, it will continue to completion, even in the dark.

2.6.3 Curing Units

UV-curing units contain one or more UV lamps, a reflector for each lamp, a means of dissipating heat from the lamps (which may be ventilating air flow), and shielding to protect people from exposure to UV.

An EB-curing unit contains an electron beam generator and shielding to attenuate x-rays that are generated during the curing process. An EB-curing unit is heavier and larger than an UV curing unit because it has more massive shielding to absorb x-rays. For both UV/EB-curing units, production rates are determined by the composition and applied thickness of the UV/EB-cured material, intensity of radiant energy in the curing unit, and amount of radiance that is absorbed. UV/EB materials cure almost instantaneously to within a few seconds when exposed to the appropriate radiant energy.

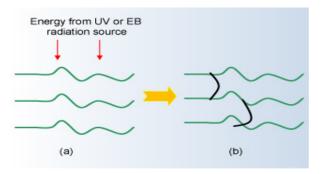


Figure 3 Interaction of UV or EB radiation with a linear polymer to develop a crosslinked network structure

When the UV or EB radiation dose increases, the molecular weight increases as well, resulting:

- A decrease of tackiness
- An increase in cohesive strength
- An increase in temperature and chemical resistance

2.7 COMPARISION OF UV WITH EB

Electron Beam	Ultra violet
1.EB technology normally require an inert	1. UV light can effect cure of coating in an
atmosphere in the processing zone, as the	oxygen atmosphere provided that the
component employed in UV curable	correct chemistry is employed
formulation are not always effective under	
EB condition	
2. The equipment incorporates	2. There is good selection of equipment
comparatively expensive high vacuum and	available to suit most types of curing
high voltage technology. Thus EB	applications

generally require comparatively high capital outlay. For this reason, it is more often employed in application where high volume through put is envisaged or where EB gives a distinct technical advantage over UV cure method.

3. EB is favored for high build or heavily pigmented system & for very fast line speed. Films up to 150 micron thick can be cured using accelerating voltage of 175 KV and line speed can be over 200 meters/ min.

4. To achieve faster line speed beam current can be increased at the same accelerating voltage, or where machine design will allow the use of multiple cathodes or double side firing can be employed with negligible effect on substrate temperature.

5. EB is favored for sensitive application due to the higher cross- link density achieved.

3. UV technology is used mainly for the curing of clear lacquers & inks. Clear lacquers are generally 4-20 microns thick, however, thicker film can be cured at the expense of line speed. Inks are pigmented system but are relatively transparent as the low film thickness is used

4. Faster Line speed can generally be achieved by employing more UV lamps or substituting more powerful lamps, to maintain the same amount of incident luminous energy per second. This obviously requires more space and generally increase the heating effect on the substrate

5. There is a limited application of UV to heat sensitive substrate because heat is generated during UV curing process.

2.8 ADVANTAGES OF ELECTRON BEAM CURING

2.8.1 Processing

Electron beam processing offers a number of other benefits over thermal curing. One of the most obvious is cost. Eight independent studies have shown potential manufacturing cost savings of 25-65%. For prototyping alone, this could rise to as much as 90-95%. This is because electron beams processing, as well as greatly speeding up curing time, cures the materials at ambient temperatures, allowing tools to be made from very low cost materials such as foam or wood. Savings are marked for complex shaped components such as engine inlets and cryogenic tanks. The stability of the resins also reduces costs because they have an almost indefinite shelf life.

Another advantage of the electron beam process is its ability to combine several different resin systems in the same curing cycle. This may not be possible with thermal curing, because different systems may need different temperatures or cure times. This is not a problem for electron beam curing because the beam of electrons is faster across the part being cured, and the 'dose' given to each area of the component can be pre-programmed. Thus, different resins are easily processed on the same line, which enables the development of new products with tailor-made crosslinking profiles to give the necessary properties in particular areas of a component.

2.8.2 Materials Properties

Electron beam cured composites often have improved mechanical properties compared to thermally cured materials, and these are retained (and sometimes improved) following thermal or cryogenic cycling. In fact, one of the key advantages of electron beam curing is that it can reduce residual internal stresses in the microstructure of composite parts. The stresses are induced during cooling and can develop within layers, owing to the low linear expansion of the fibers compared to the resin, or between layers. Such stresses can limit composite part design. However, electron beam curing reduces such stresses because processing temperature can be ambient or even lower. This is very beneficial for producing cryogenic tanks, which must hold fluids at temperatures of -200 to -255°C.

Conventional thermal curing is carried out at 150 to 200°C, and this large difference can causes large stresses, which are eliminated using the electron beam technique.

2.8.3 Environmental and Health

The electron beam technique also offers environmental and health benefits. The amount of volatiles produced using this technology is reduced significantly, with the resins typically producing 0.1% or less volatiles. Stack emissions of volatile chemicals from electron beam curing can be as low as 1% of the emissions from thermally cured resins. In addition, chemical crosslinking agents for thermosetting resins, which can be toxic and carcinogenic, can be eliminated.

2.9 EQUIPMENT USED

2.9.1 Ultraviolet Curing

Even if the radiation-cured coatings present many advantages and are applicable to a wide range of product, it's progress is slowed down by the high materials cost and capital investment. Typical UV equipment consists of:

1. Lamp: the electrical energy supplied to the bulb is converted into UV energy inside it

- Type of lamp (electrode/ electrode less/ pulsed/ spot)
- Spectral output of lamp ("H"/'D"/'V")
- Length of lamps (long lamps tend to sag)
- Reflector (elliptical/ parabolic/ dichroic)
- Focused/unfocused reflectors
- UV lamp output (200 to 600 watts/inch) (H bulb emits fewer photons than a D bulb at the same watt/inch rating
- Curing atmosphere (N₂/ ambient/dry air)
- Required UV exposure (dwell time)
- Acceptable window of equipment operation

2. Lamp housing: reflects and focuses the UV energy generated by the lamp (delivers the UV to the substrate)

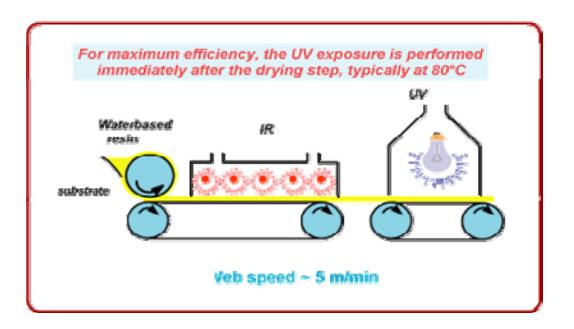


Figure 4 Example of process for UV-curing of waterborne PUA coatings

2.9.2 Electron Beam Curing

Typical EB equipment consists of:

- i. Electron accelerator
- ii. Optical system focuses the accelerated electrons

The faster or more energetic an electron is, the deeper it will penetrate into a given substrate, the source is usually an electrons filament or cathode. The cathode is generally heated within a high vacuum chamber and emits electrons as its temperature is raised. The cathode heating and subsequent electron acceleration must take place inside vacuum, otherwise the electrons produced would scatter rapidly on collision with molecule in the air, resulting in no effective increase in their velocity.

Various types of equipment are available to produce high speed electrons, which are as follows:

2.9.2.1 Scanned beam type

It is similar to a television cathode ray tube. The electron is emitted from a heated spiral point cathode with a diameter of 5 mm which is contained with in a high vacuum. The focused beam is scanned across the web using an electromagnetic deflection system with a frequency between 50-200 Hz.

Delivered dose is determined by the dwell time of the beam on the substrate. The accelerator voltage is between 150 & 280 KV, depending upon the application involved.

Beam cured is controlled by the temperature of the cathode & because this is non linear in relationship a very sophiscated regulation device is required to bring about a rapid response linear relationship, for positive beam current control.

The beam produced passes into the process zone through a 12-15 micron thin titanium foil window which is supported & cooled from the vacuum side.

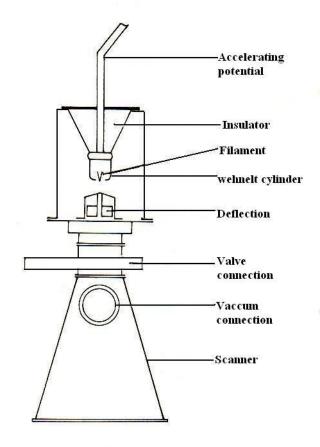


Figure 5 Scanner type electron processor

2.9.2.2 Linear cathode type

This design of accelerator is also known as the electro curtain design. It is the design of accelerator which has probably seen the widest adoption in industry, certainly in the area of curing surface coating. The linear cathode system works in the following way:

Electrons are drawn from the cathode (c) which is a little longer than the width of the process zone, by the application of a relatively low voltage to the grid (G). This grid current is used to control the number of electron per second extracted from the filament. The strip or curtain of electron (B) is then accelerated by the electric field which rises from the large negative potential on housing terminal (T) to the outer wall of the vacuum

chamber (w). This voltage can be 100 KV to 300 KV. All the possible energy is imparted into the electron in this space. The beam of accelerated electron exits the high chamber through the thin titanium foil window (f), (which is supported on the vacuum side & cooled continuously) into the process zone to contact the product (p) which is to be irradiated.

The electrons are moving at virtually moving at the speed of light as they strike the window and consequently only lose about 10-15% of their energy in the foil. As the beam of electron moves into the air path they tend to scatter with the beam becoming wider. For this reason, the product to be processed is place as close as possible to the window so as to reduce energy losses to negligible level.

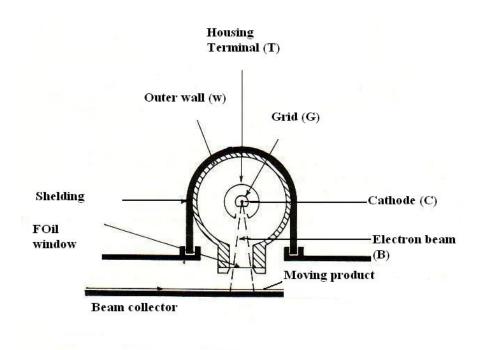


Figure 6 Electrocurtain type electron processor

2.9.2.3 Broad beam type

In this, the significant difference is that the broad beam system employ multiple linear cathodes 300mm long sited in the web direction arranged in modules utilize a common extraction grid mounted between the filament grid modules & the out put window. The dose rate capacity is thus increased by a longer dwell time of product under the electron beam.

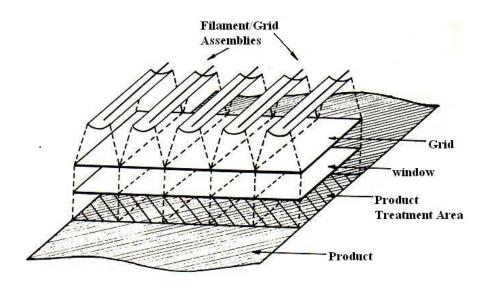


Figure 7 Broad beam processor schematic

2.10 RAW MATERIALS

As with coating, formulation variables are critical to the processing and performance characteristics. Practical radiation curable materials are composed of more than only a single reactive monomer. Commercial coatings normally contain the following:

- 1. Oligomer a base resin reactive material
- 2. Photoinitiators (UV coatings only)
- 3. Monomers (Mono, di and multifunctional)
- 4. Additives (Stabilizers, Adhesion promoters, Pigments, etc.)

2.10.1 Oligomers

They are low molecular weight compounds. The molecular weight varying from 400 to 7000. It is a base resin reactive material.

The oligomers which undergo free radical polymerization are as follow:

- 1. Epoxy Acrylate
- 2. Urethane Acrylate
- 3. Polyester Acrylate
- 4. Polyether Acrylate
- 5. Acrylic Acrylate
- 6. Miscellaneous Resins

1. Epoxy acrylate

Epoxy acrylate is the reaction product of Bisphenol A epoxide with acrylic acid, e.g. Bisphenol A – epoxy resin reacts with acrylic acid or methacrylic acid to provide unsaturated terminal reactive group. The versatility of the backbone structure provides a wide range of chemical coating properties. The epoxy base oligomers may be viscous. The acrylic acid/epoxy reaction to make Bisphenol-A-diacrylate destroys any ingredient such as epichlorohydrin which is used to make bisphenol-A epoxy starting raw material. The chemical structure of Bisphenol-A, diepoxide reacted with acrylic acid to form a Bisphenol-A epoxy diacrylate is given in fig- 10. Acrylated epoxies are fast curing and abrasion & chemical resistant but have poor flexibility & will yellow when exposed to out door weathering conditions.

Epoxy acrylates are dominant oligomers in the radiation curable coatings market. In most cases epoxy acrylates do not have any free epoxy groups left from their synthesis but react through their unsaturation. Within this group of oligomers, there are several major sub classifications: Aromatic difunctional epoxy acrylates, acrylated oil epoxy acrylates, Novolac epoxy acrylate, aliphatic epoxy acrylate, and miscellaneous epoxy acrylates.

Properties

- High gloss, high hardness
- Fast cure
- High viscosity
- Yellowing on out door wetting



Figure 8 Bisphenol A epoxide with acrylic acid

Aromatic difunctional epoxy acrylates

Aromatic difunctional epoxy acrylates have very low molecular weight, which gives them attractive properties such as high reactivity, high gloss, and low irritation. Common applications for these resins include overprint varnishes for paper and board, wood coatings for furniture and flooring, and coatings for compact discs and optical fibers. Aromatic difunctional epoxy acrylates have limited flexibility, and they yellow to a certain extent when exposed to sunlight. The aromatic epoxies are viscous and need to be thinned with functional monomers. These monomers are potentially hazardous materials.

Acrylated oil epoxy acrylates

Acrylated oil epoxy acrylates are essentially epoxidized soya bean oil acrylate. These resins have low viscosity, low cost, and good pigment wetting properties. They produce relatively flexible coatings. Acrylated oil epoxy acrylates are used mainly in pigmented coatings or to reduce cost.

Epoxy Novolac acrylates

Epoxy Novolac acrylates are speciality products. They are mainly used in the electrical/electronics industry because of their excellent heat and chemical resistance. However, they provide rigid coatings with relatively high viscosity and high costs.

Aliphatic epoxy acrylates

Aliphatic epoxy acrylates comprise of several varieties. They are available as difunctional, trifunctional etc. The difunctional types have good flexibility, reactivity, adhesion, and very low viscosity. Some difunctional types can be diluted with water. The trifunctional or higher types have moderate viscosity and poor flexibility but excellent reactivity. Aliphatic epoxy acrylates have higher cost than the aromatic epoxy acrylates and are generally used in niche applications.

Miscellaneous epoxy acrylates

Miscellaneous epoxy acrylates consist mainly of oligomers with fatty acid modification. They provide good pigment wetting properties and higher molecular weight but lower functionality than other aromatic epoxy acrylates. They are used in printing inks and pigmented coatings.

2. Urethane acrylate

Urethane acrylates are produced by reacting polyisocyanates with hydroxyl alky acrylates, usually along with hydroxyl compounds, to produce the desired set of properties. Urethane acrylates are the most expensive of the acrylates.

Molecular weight may be changed from only a few 100 to 10000. Typical urethane acrylate molecular weight is 8000 to 5000.Urethane acrylate is prepared by the reaction of a hydroxyl group of a monomer such as hydroxyl propyl acrylate with an isocynate group e.g. radicals when hydroxyl compound such as polyester, polyether & polyols which contains more than one hydroxyl group per molecule are employed then chain

lengthening occur. They are many different types of urethane acrylate oligomers having variation in the following parameters:

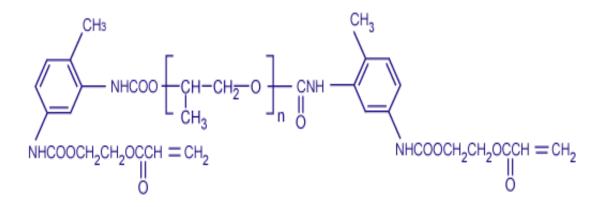


Figure 9 Typical urethane acrylate oligomer

Functionality

It varies from one to six. Lower functionality results in lower reactivity, better flexibility, and lower viscosity. Monofunctional urethane acrylates are low viscosity, speciality products used to improve adhesion to difficult substrates and to improve flexibility. High functionality products (4 and higher) have niche applications as well. They are used to improve reactivity, scratch resistance, chemical resistance, and other physical properties. Because of their high viscosity, they are generally blended with other resins.

Isocyanate

Four types of isocyanates are used for urethane acrylates. Mono isocyanates are used for monofunctional acrylates only. Diisocyanates are the most widely used and can be divided into aliphatic di isocyanates and aromatic di isocyanates. The incorporation of an aromatic diisocyanate makes the resulting coating harder and abrasion resistant. The higher cost aliphatic di isocyanates are slightly more flexible. However, they are non-yellowing. Aliphatic urethane acrylates are used for topcoats, optical fibers, flexible packaging, etc. Polymeric isocyanates are used for higher functionality urethane acrylates.

Polyol

The polyol is the backbone of the urethane they are essentially polyether or polyester with functionality ranging from two to four. Polyether urethane acrylates are generally more flexible, have lower costs, and have slightly lower viscosity. Polyester urethane acrylates have less hydrolytic stability but are non-yellowing.

Molecular weight

For di & tri functional urethane acrylate, the polyol modifier determines this property. Some of the variables that are present in the design of urethane acrylate include the following:

- Degree of unsaturation : This is controlled by the selection of hydroxyl monomer, molecular weight of the pre- polymer, isocyanates content and the nature & level of the reactive monomer
- 2. Nature of the hydroxyl monomer
- 3. Nature of the di- isocynate
- 4. Viscosity & molecular weight i.e. the higher the viscosity the greater the molecular weight. This affects the amount of monomer to be added in the coating and this effects application viscosity, adhesion, flexibility etc

Properties

Resin type	: Urethane type
Functionality	: 2
Molecular weight	: 1550
Appearance	: clear liquid
Colour	: gardner3
Viscosity at 40°c	: 1000p
Flash point	$: >125^{\circ}C$

3. Polyester acrylates

The wide choice of polyester acrylates available enables a broad range of demand on the coating to be met. They are generally low viscosity resins that require no reactive diluents. Polyester acrylates provide performance properties between those of urethane acrylates & epoxy acrylates. A disadvantage of some types of polyester acrylates is their irritancy. This is particularly true for low molecular weight, highly reactive resins. Polyester acrylates vary in functionality, chemical backbone & molecular weight. The chemical backbone has a large influence on properties such as reactivity, colour stability, hardness, reactivity etc. Typically, the higher the molecular weight, higher is the flexibility & viscosity and the lower is the reactivity.

4. Polyether acrylate

Polyether acrylates have the lowest viscosity of the acrylate resins and are typically used with very little monomer or reactive diluents. They are made by esterifying polyether with acrylic acid. They generally have high flexibility but relatively poor water and chemical resistance. To overcome these drawbacks, polyether acrylates are mostly used in combinations with other oligomers or monomers.

Polyether acrylates can be made more reactive by adding amine groups. The water resistance and chemical resistance of amine modified polyether acrylates are much better than of unmodified polyether acrylates. However, they still lack the flexibility and toughness of urethane acrylates. The main applications of these resins are in wood coatings for furniture and paper coatings.

5. Acrylic acrylate

Like urethane acrylates, the chemistry of acrylic acrylates is very versatile and there are many variations available to the formulation. These resins are often used because of their good adhesion to difficult substrates such as low surface energy plastics.

6. Miscellaneous resins

Miscellaneous oligomers are generally speciality products that typically comprise melamine acrylates, silicone acrylates, etc. Other types of radiation curable resins include unsaturated polyesters dissolved in styrene or acrylics. More recently, polyester resins have appeared in the market in the form of non-acrylic vinyl ether blends.

Epoxy and glycidyl ethers are the main resins used in cationic polymerization systems. They provide low shrinkage and good adhesion. Their major disadvantage is poor reactivity which can be improved somewhat by the addition of vinyl ethers. Cationic systems occupy small and niche markets, but they are expected to have a growth rate somewhat higher than the growth of overall radiation curable coatings

2.10.2 Photoinitiators

The photoinitiator is an ingredient that absorbs light and is responsible for the production of free radicals in a free radical polymerized system or cations in a cationic photo initiated system. The photoinitiators are usually added to the reactive coating formulations in concentration ranges from less than 1 to 20 weight percent based on the total formulation. The absorption bands of the photoinitiators should overlap the emission spectra of the various commercial UV Bulbs (H bulb, D bulb, etc.). There are two general classes of photoinitiators:

- 1. Photoinitiators undergo a unimolecular bond cleavage upon irradiation to yield free radicals.
- 2. Photoinitiators undergo a bimolecular reaction where the excited state of the photoinitiator interacts with a second molecule (a co initiator) to generate free radicals. The choice of photoinitiator is determined by:
- The radiation source, the film thickness
- The pigmentation
- The types of base resin employed

It is widely used in radiation cured coating

Some of the photoinitiators used are listed as below:

- 1. Benzoin butyl ether
- 2. Benzil ketals

2.10.3 Monomers

Monomers are of three types

- 1. Monofunctional monomers
- 2. Difunctional monomers
- 3. Multifunctional monomers

There are four major parameters that contribute to the monomer characteristics which are shown in table 1

Monomer diluents are mono functional monomers primarily used to lower the viscosity of the uncured material and to facilitate application. Crosslinking monomers are difunctional or multifunctional monomers containing two or more reactive sites. They crosslink the polymer chains by forming links between oligomer molecules, since there are so many monomers available, it is important to keep in mind some general guidelines. There are four major parameters that contribute to the monomer's characteristics: functionality, chemical backbone, chemical structure and molecular weight. These are defined and related to coating performance in Table 1

Table 1 Parameters of Monomer characteristic

Parameter		Characteristics
Functionality		Higher the functionality, higher the
		reactivity. The lower the functionality, the
		lower the viscosity. The lower the
		functionality, the lower the polymerization
		shrinkage.
Chemical	Hydrocarbon	Low surface tension, good flexibility, low
	type	yellowing and good weathering properties,
backbone		excellent water resistance.
Chemical	Ether type	Have a higher polarity than hydrocarbons,
backbone		so are better reactive diluents. More
		reactive than hydrocarbons, but poor in
		non-yellowing and weather resistance.
Chemical	Alkoxylated	Low irritancy, higher polarity than
backbone		hydrocarbons, higher reactivity despite
		high molecular weight.
Chemical	Propoxylated	Have low surface tension in combination
backbone		with lower viscosity than hydrocarbon
		monomers, good wetting and adhesion to
		difficult substrates.
Chemical structu	re	Can be cyclic, branched, or linear. The
		main effect is on Tg which generally
		follows: cyclic > branched > linear.
Molecular weight		The lower the molecular weight, generally
		the lower the viscosity and the higher the
		reactivity and the higher the Tg. In
		changing chemical backbone and structure
		the molecular weight will also change.

Some of the monomer used in radiation cured coating includes

Tripropylene glycol diacrylate (TPGDA): This difunctional monomer has low viscosity & gives reactivity. This is used in formulation for wood.

Trimethylol propane ethoxylate acrylate: This has a viscosity of approximately 380 cps & offers high reactivity. It is used as one of the monomer in coat to enhance the cure rates.

Monofunctional Monomers

Monofunctional monomers are low molecular weight materials that chemically incorporate into the cured coating rather than volatilize into the atmosphere as is common with solvent diluents.

Monofunctional diluents are chosen on the basis of providing good wetting, flexibilizing and low shrinkage. They generally provide soft, thermoplastic films because of their linear and uncrosslinked nature.

Phenol Ethoxylate monoacrylate

Figure 10 Example of monofunctional acrylate

Difunctional Monomers

Difunctional monomers act as crosslinking agents and thus, crosslink the polymer chains as the film is cured, forming links between oligomer molecules and other molecules in the formulation. Monomers of this type are able to cure very rapidly providing low volatility, high reactivity and good flexibility. Tripropylene glycol diacrylate

$$H_2C=CH-C-C+CH_2-O-C+CH_2-CH_2$$

Figure 11 Example of difunctional acrylate

Multifunctional Monomers

Multifunctional monomers act as crosslinking agents and thus, crosslink the polymer chains as the film is cured, forming links between oligomer molecules and other molecules in the formulation. Multifunctional monomers are able to cure very rapidly providing high cross-linking density, fast cure response, good flexibility and low skin irritation.

Trimethylolpropane ethoxylate triacrylate

 $C_2H_5 - C - CH_2O - CH_2CH_2O - CH - CH - CH_2$

Figure 12 Example of trifunctional acrylate

2.10.4 Additives

- 1 Monomer diluents
- 2. Stabilizers
- 3. Oxygen scavengers
- 4. Adhesion promoters
- 5. Pigments

Monomer diluents

Monomer diluents are primarily used to lower the viscosity of the uncured material and to facilitate application. They are usually low molecular weight, monofunctional materials that chemically incorporate into the cured coating rather than volatilize into the atmosphere as is common with solvent diluents. Monomer diluents are chosen on the basis of providing good solvency, effectively reducing the viscosity of the oligomer without excessively retarding cure rate. Certain diluents will contribute to the physical properties of the coating. However, generally they provide soft, thermoplastic films because of their linear and uncross linked nature

Stabilizers

Stabilizers are common additives included in coatings to prevent premature polymerization resulting in viscosity increase, gelation in storage, or premature curing that occur due to low levels of light exposure. These compounds act as scavengers for free radicals. They neutralize the free radicals before they have a chance to start the chain reaction leading to polymerization Light stabilizers, such as hindered amine light stabilizing (HALS) compounds, also protect the cured coating from exposure to direct sunlight. These products are used to provide coatings with good outdoor stability, color retention, and non-yellowing properties.

Oxygen scavengers

Oxygen scavengers may be required as oxygen inhibits the curing of acrylates by quenching the photoinitiator or by scavenging free radicals. Scavenging produces stable species that slow down the cure rate but also can degrade the properties of the cured coating. Other methods of oxygen inhibition are nitrogen blanketing (a process called "inerting") or by speeding up the cure by using higher intensity lamps and by varying the initiator type and concentration. The specific coating determines the propensity for oxygen inhibition and the degree of sophistication needed to counteract it.

Adhesion promoters

Adhesion promoters are used to provide good coating adhesion to such substrates as glass, hard plastics, and certain metals such as brass. These are specialized materials (e.g., organosilanes), which have the ability to promote adhesion to a substrate material, and at the same time form a part of the coating's polymer network.

Pigments

Pigments generally inhibit UV curing to some degree since the pigments absorb and/or scatter UV radiation. This interferes with the ability of the photoinitiator to absorb the light energy required to initiate the polymerization reactions. Thus, the majority of commercial radiation curable coatings is clear or contains silica. Investigators have shown that free radical formulations that are white pigmented and color pigmented can be UV cured by selecting the proper photoinitiator or photoinitiator combinations. Colorizing pigments have only a minor effect on the cure rate of high energy electron beam (EB) coating formulations.

2.11 CURED COATING PROPERTIES

2.11.1 Gloss

UV/EB curable coating generally gives high gloss transparency. Epoxy acrylates reach high gloss value. The high gloss effect is related to low chemical film shrinkage compared to high physical shrinkage through emissions of solvents based system.

2.11.2 Chemical resistance

It is an important property of coatings. Resistance to chemicals like MEK, acetone or household products like tea, coffee, hot water, etc. can be checked. To increase chemical resistance, very often acrylates with double densities (high acrylate functionality & low molecular weight) are used. The higher tri dimensional cross linking will lead to higher

molecular weight, tightly crosslinked and chemically resistant films. In general epoxy acrylate gives very good results with respect to chemical resistance.

2.11.3 Scratch resistance

Radiation curable coatings are generally hard & scratch resistance compared to conventional systems. Scratch resistance is a function related to hardness of first microns of coating, so improving scratch resistance requires increasing the coating surface crosslink density. This can be achieved by:

- 1. Reducing surface oxygen inhibition by curing under inert atmosphere
- 2. Using higher functional acrylates
- 3. Using amine modified oligomers
- 4. Using abrasive fillers

2.11.4 Abrasion resistance

Abrasion resistance is an important performance property of any kind of coating. Abrasion resistance is also one of the most complex properties to attain, as it is both a surface and subsurface property.

Abrasion can be checked by using taber abrasion. In this test coating is given predetermined number of cycles at fixed load using a standard abrasive wheel & the weight loss is reported. For example CS-17 wheel can be used to give 500 cycles at 1000 gms load. Urethane acrylate gives high abrasion resistance followed by medium viscosity polyester and then flexible epoxy acrylates.

2.11.5 Glass transition temperature (Tg)

It is the temperature at which the coating changes from hard abrasion resistance coating to a soft, rubbery material. Usually higher the Tg the greater the coating hardness. The Tg of coating can be determined using Differential Scanning Calorimeter.

3.1 EMITTED POLLUTANTS FROM UV/EB-CURED MATERIALS

UV/EB-curing is a very low emitter of air pollutants, but some emission does occur. Such as VOC, fine particulate, HAP, odors, ozone and NO₂

3.1.1 VOC Emissions

UV/EB-cured coatings have a reputation for being VOC free, but they actually do emit some cure volatiles that may be VOC. Although VOC emissions are extremely low from many applications using modern UV/EB materials (i.e. emissions can be less than 1 % of the weight of the coating) cure volatile emissions for a typical application usually are in the 1-5 % range. (Note that no solvent diluents are used in the typical case.) However, for a small number of applications, cure volatile emissions can approach 10 % of the applied film weight. Even at the 10 % level, uncontrolled VOC emissions are no worse than that of a conventional coating with good emission capture and control technology.

3.1.2 UV/EB-cured materials

UV/EB-cured materials are ready to use as provided by the manufacturer. The one exception may be adding a solvent (VOC or water) to adjust viscosity in some spray or special applications. UV/EB-cured materials will not dry up unless subjected to sufficient UV/EB radiant energy. If diluents are present, uncured UV/EB coatings may simply become more viscous as the diluents evaporate. However, normally VOC diluents are not present.

Acrylates have been measured at a concentration of approximately 10 ppm in the space immediately above a coating during cure. Concentrations of VOC in the same space were even lower. Concentrations in a subsequent emission stream would tend to be even lower as a result of mixing and dilution with air in the exhaust stream. Such small emission concentrations are the desired goal of add-on emission control technology used with conventional processes, but they can be achieved by using UV/EB-cured coatings, inks and adhesives without an add-on emission control device.

3.1.3 Ozone and NO₂ Emissions

Each form of ionizing radiation can ionize oxygen and generate ozone. Also, there is suspicion that NO₂ can be generated, too. However, total emission of ozone and NO₂ are relatively small and should not significantly affect concentrations of these pollutants in the atmosphere. Equipment manufacturers have reduced the potential for ozone and NO₂ generation from processes that use UV/EB radiant energy. Nitrogen blankets exclude air from the polymerization areas and, therefore, exclude oxygen as a reactant in these areas. However, air ventilation is used to remove heat from UV lamps; therefore, these pollutants still can be generated. Quartz filters that strongly absorb wavelengths below 260 nm (the wavelengths that form ozone) can be used to reduce ozone generation.

3.2 WORKERS HEALTH AND SAFETY

There are three major worker health and safety issues that need to be addressed

(1) Potential exposure to UV/EB radiation

(2) Potential exposure to hazardous components that are part of UV/EB-cured materials

(3) Hygiene to protect workers. This section will discuss all of these issues and significant changes and improvements made over the past ten years to minimize potential hazards

3.3 HEALTH AND SAFETY CONCERNS DOES UV/EB RADIANT ENERGY PRESENT

UV radiant energy is ultraviolet light. This is the same type of ultraviolet light that is received from the sun. It cannot penetrate the skin, but can cause sunburn and tanning of the top layer of skin or can cause eye irritation. When properly shielded, UV does not harm or cause such problems.

EB radiant energy consists of high velocity electrons and subsequent x-rays. Electrons are generated in an accelerator or gun that projects them toward the end substrate. Electrons generate x-rays as their speed is retarded in these materials, and by any molecules between the EB source and the target film and substrate. Manufacturers of this EB equipment install shielding to reduce x-radiation to below background level.

3.4 PRECAUTIONS

Direct skin contact with all coatings should be avoided. Always hands wash thoroughly before using restroom facilities. Barrier creams should be applied to clean skin before exposure and not applied after exposure. Barrier cream should subsequently be reapplied after washing hands and before returning to work. Barrier creams can protect against material that might penetrate through defects in the protective equipment. Typically, fabric or non-woven long sleeved and full leg clothing or coveralls are worn. Rubber gloves should always be worn when direct contact with radiation curable materials is possible. It is appropriate to use the same hygiene for UV/EB materials that you would use for a solvent, a corrosive chemical, or a toxic chemical. Wearing protective clothing for a full shift may also require that air temperature be controlled to allow workers to remain comfortable while working.

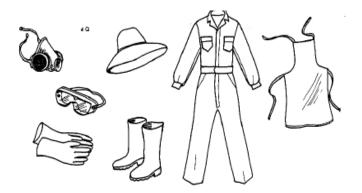


Figure 13 Examples of safety equipment

Chapter 4

The Experimental work was carried out with the selection of the wood, preparation of the coated sample followed by the EB curing of the coating. Final results were analyzed on the basis of the properties of the cured composition on wood.

The experimental work carried out this study includes:

- Preparation of wood sample for coating
- Selection of raw materials
- Development of coating composition
- Application of coating composition on wood
- > Curing of coating composition by EB irradiation process
- Evaluation of irradiated wood samples

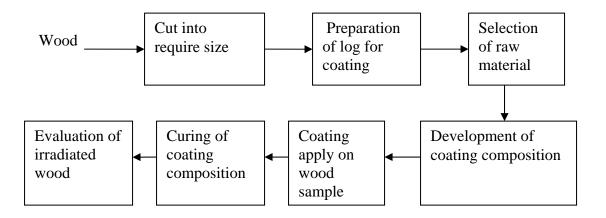


Figure 14 Flow chart for wood coating

4.1 PREPARATION OF WOOD SAMPLE FOR COATING

Three different types of wood were selected for coatings, i.e. Teak, Kail, Kikar. These woods were cut into different size. Sand paper was used to make surface of the cut wood samples, smooth & uniform by buffing. Coat of clay, chock powder etc, was applied on the wood sample to fill the pores. Sample was again buffed with fine sand paper after drying until the desired surface is obtained.

4.2 SELECTION OF RAW MATERIALS

The Acrylic compounds and their substitutes were selected as raw materials. Acrylics are known for their best curing, adhesive and weathering properties. They posses resistant to moisture attack. Diacrylates are better known for the better mechanical strength, fine gloss at an economic rate. The diluent used was Tripropylene glycol diacrylate.

S.No	Raw Material	Composition	Use
1	Photomer 3016F	Epoxy diacrylate	Oligomer
2	Photomer 4017F	Hexane –diol diacrylate	Reactive Diluent
3	Photomer 4226F	Dipropyleneglycol diacrylate	Reactive Diluent
4	Photomer 4061F	Tripropyleneglycol Diacrylate	Reactive Diluent

Table 2 Raw material for wood coating composition

4.3 PREPARATION OF COATING COMPOSITION

A number of coating composition were developed using oligomers & reactive diluents in varying ratios. The procedure was repeated for the same oligomer using the same different diluents in the same quantity. All these compositions were cured by electron beams of different intensities. The best results were found using 400 KGy as shown in table 3

Batch No.	Oligomer	E	Diluent (phr)		
	3016	4017	4017 4061 4226		Dose 400
					(KGy)
ABC 1	100	20			400
ABC 2	100		20		400
ABC 3	100			20	400

Table 3 Composition of wood coating

4.4 APPLICATION OF COATING COMPOSITION ON WOOD

The coating composition was applied on the wood sample manually by roller coating technique. Some coating material was pored along one edge of the wood sample & it was spread uniformly by using the glass rod. Excess material is removed by the gloss rod. Spray & brushing coating technique were also tried for apply coating composition on wood but this process was not successful because of high viscosity of the coating composition.

4.5 CURING OF COATING COMPOSITION

The coated wood samples were exposed in radiation zone and checked at minor dose i.e. 100, 200, 400 KGy. The samples were cured at 400 KGy

4.6 EVALUATION OF IRRADIATED WOOD SAMPLES

4.6.1 Pencil Hardness

The pencil hardness was evaluated according to ASTM: D 3363. This test method covers a procedure for rapid inexpensive determination of the film hardness of an organic coating on a substrate in terms of drawing leads or pencil leads of known hardness.

A coated panel is placed on a firm horizontal surface. The pencil is held firmly against the film at 45 angle and pushed away from the operator in a ¹/₄-in (6.5) mm stroke. The process was started with the hardest pencil and continued down the scale of hardness to either of two end points: one, the pencil that will not cut into or gouge the film (pencil hardness), or two, the pencil that will not scratch the film (scratch hardness). A set of calibrated drawing leads (preferred) or equivalent calibrated wood pencil meeting the following scale of hardness:

<u>6B- 5B- 4B- 3B- 2B- B- HB- F- H- 2H- 3H- 4H- 5H- 6H-</u> Softer Harder

The difference between two adjacent leads shall be considered one unit of hardness.

Procedure

- 1. For wood pencils, removed approximately 3/16 to $\frac{1}{4}$ in (5 to 6 mm) of wood from the point of each pencil using a draftsman – type mechanical sharpener, being careful to leave an undisturbed, unmarked, smooth cylinder of lead. Holding the pencil holder at an angle of 90^{0} to abrasive paper , rubbed the lead against the paper maintaining an exact angle of 90^{0} to the abrasive paper until a flat, smooth and circular cross section was obtained, free of chips or nicks in the edge of the cross section. The desired edge may be obtained by cementing the abrasive paper to flat motor-driven disk a uniform flat lead end may be obtained more reproducibly.
- 2. Placed the coated panel on a level, firm, horizontal surface starting with the hardest lead, hold the pencil or lead holder firmly with the lead against a 45°

angles and push away from the operator. A sufficient uniform pressure exerted downward and forward either to cut or scratch the film or to crumble the edge of the lead. The length of the stroke is ¹/₄ in. (6.5mm).

- 3. Repeated the process down the hardness scale until a pencil is found that will not cut through the film to the substrate.
- Continued the process until a pencil was found that will either cut through or scratch the surface of the film. Recorded each end point for gauge and scratch hardness.

4.6.2 Resistance to steam

The stain resistance of the wood sample was evaluate according to the EN 438-2 : 1991.A specimen from the sheet under test is held in place over the neck of a flask containing boiling water, so that the decorative surface of the specimen is exposed to the steam. After one hour, the specimen is removed and allowed to recover for 24 hour in normal ambient condition before examination for any change in appearance.

Materials

- 1. Erlenmeyer flask, wide- necked, of capacity 250 ml
- 2. Specimen holder and heat screen
- 3. Non Fibrous filter paper
- 4. Hand lens with 6x magnification
- 5. Electric hot plate hot plate or other suitable heat source

Procedure

The wood sample of 100 mm x 100 mm was taken. Placed approximately 200 ml of water in the flask and brought it to the boil on the electronic hot plate. Placed the heat screen in position around the neck of the flask, and placed specimen, decorative face down, centrally over the mouth of the flask and fixed it in position by the wire specimen holder. After the decorative face has been exposed for 1 hour to the steam from the boiling water, removed the specimen and use the non- fibrous filter paper to remove excess water from the surface of the specimen.

Allowed the specimen to recover for 24 hours in normal ambient condition and then examined the central area of the specimen with naked eye, corrected if necessary, and under 6 x magnification using the hand lens for any change in appearance.

4.6.3 Resistance to staining

The stain resistance of the wood sample was evaluating according to the EN 438-2: 1991. Specimens were left in contact with a series of staining agents which are likely to be encountered in every day use. The time and condition of contact are specified for each staining agent. At the end of the specified contact period, the specimens are washed and examined for residual surface marks.

Apparatus and materials

- 1 Glass covers (for example watch glass) to restrict evaporation
- 2 Thermometer, range 100° C
- 3 Hot plate or other suitable heat source
- 4 Horizontal inspection surfaces
- 5 Wetting agent
- 6 Ethanol, 95 %, (volume fraction), acetone, NaOH, H_2O_2 , etc.
- 7 Soft clean cloth
- 8 Hard nylon bristle brush

Procedure

The specimens were initially at ambient temperature. Applied a small quantity (2 or 3 drops) of test materials to two specimens. The test material should be at the temperature specified in table. Covered the material on one of the two specimens with a glass cover. After the specified contact time has elapsed, removed the staining material with a suitable detergent, then washed with water containing a suitable wetting agent, and finally with ethanol or other solvents as required to clean the surface. A suitable brush (see 8) may be used to remove staining material from textured surface.

One hour after washing, placed the specimen on the inspection surface and viewed it from various angles at a distance of 400 mm.

Test materials	Test condition	Contact time
Group 1	Apply test material at	
1. Acetone	ambient temperature	
2. Hand cream		16 to 24 hours
3. Animal, vegetables fats		
and oils		
Group 2	First two at 80 [°] C Temp	
1. Coffee (120gm coffee per		
liter of water)		16 hours
2. Milk		
3. Cold beverages		
4. Ball point inks	Last two at ambient temp	
Group 3		
1. Sodium hydroxide		
2.NaOH (25% solution)		10
3.Hydrogen peroxide H2O2	Apply test material at	10 min
(30% solution)	ambient temperature	
4.Concentrated vinegar		
(30% acetic acid)		

Table 4 List of staining material

4.6.4 Gloss

The gloss of coated wood sample was evaluated according to ASTM: 2457-90. The wood coated sample was taking. The samples were proper clean with a tissue paper. Now the gloss measure by the Novo Gloss meter. The gloss meter was placed on the coated wood

sample at different places. Noted down the reading, considered the maximum four reading & took an average of values of gloss.

4.6.5 Water absorption

The water absorption of the wood sample was evaluated according to the IS: 2380 (part-XVI). The required test specimens was cut to the specified size (30 x 30 cm) subject to a tolerance of ± 2 mm on the width and length and should be prepared and conditioned. All the four edges should be smoothly and squarely trimmed.

Procedure

Mass and Volume of Test Specimen

After conditioning the specimen weighed to an accuracy of not less than ± 0.2 % and the width, length and thickness measured to an accuracy of not less than ± 0.3 %. The volume of the specimen computed from these measurements.

Submersion in water

The specimen then was submerged horizontally under 25mm fresh clean water maintained at temperature of $27 \pm 2^{\circ}$ C. The test specimen separated by at least 15mm from each other from the bottom and sides of the container. After a 2 hour submersion, the specimen was suspended to drain for 10 minutes, at the end of which time the excess surface water was removed and the specimen immediately weighed. The specimen was submersed for an additional period of 22 hour and the above weighing procedure repeated.

Moisture content

Each specimen weighed to an accuracy of not less than ± 0.2 %. After submersion, the specimen dried in a ventilated oven at $103 \pm 2^{\circ}$ C until the mass was constant to ± 0.2 % between two successive weighing made at an interval of not less than 1 hour. The

moisture content of the specimen calculated after conditioning and after 2 and 24 hour submersions as per the formula given below:

$$M_1 - M_2$$
Moisture content = $\frac{M_1 - M_2}{M_0}$ X 100

Where

$$M = \text{Initial mass}$$
$$M = \text{Oven dry mass}$$

Density

The density of the test specimen may be calculated by this formula:

$$Mass of test specimen in g x 10^{6}$$

$$Density = ----- Kg/m^{3}$$

$$Length (mm) x Width (mm) x Mean thickness (mm)$$

4.6.6 Resistance to Cigarette Burns

The resistance of cigarette burn of the wood sample was evaluated according to the EN 438- 2: 1991. In this test wood sample were taken. Two brand cigarettes like Red & white, and Ultra mild was used. Each with a mass of 1.0, 1.1 gm. For a length of 70mm and the tobacco evenly distributed over its length. Ignited one cigarette from each of the brand and let each one burn to consume a length of approximately 10 mm. placed one of the burning cigarettes in full length contact with the horizontal surface of the specimen of in a draught free area. So as the glued seam of the cigarette is not in contact with the specimen. Allowed the cigarette to continue burning until a further 20 mm length was consumed. If the cigarette was smoked out in between, the test was repeated. The sample was wiped off with a tissue paper or soft cloth moistened with alcohol. The surface was examined such as discolouration, cracks or blisters.

4.6.7 Dry heat resistance

The Dry heat resistance of the wood sample was evaluated according to the EN 438- 2: 1991. Coated wood samples were taken. A flat bottom, open aluminum vessel of 8cm bottom diameter, 14 cm top diameter and 6 cm height was taken. The vessel was filled with silicon at level 15 mm from the top. The temperature of the silicon oil was raised to 185°C. This was measured by the thermometer which was dipped in the silicon oil. The silicon oil was continuing stirred to maintain the uniform temperature of the silicon oil. The temperature was raised 185°C. The vessel was removed from the heating plate. The temperature fell to 180°C. The vessel was placed on the wood sample immediately for 20 minute. After 20 minute the vessel was removed and the wood sample was cooled for 45 minute. After 45 min the sample was examined such as discolouration, loss of gloss, shrinkage.

5.1 RATING FOR STAIN RESISTANCE

Different wood samples were prepared using selected composition under investigation. These samples were cured prior to their characterization. Firstly, rating for stain resistance is reported here

- 1: Tacky rim
- 2: Black stain
- 3: Dark brown stain
- 4: Light brown stain
- 5: Yellow stain
- 6: pink stain
- 7: White rim
- 8: Faint rim
- 9: Glossy mark
- 10: No Change

5.2 RATING FOR STEAM, DRY HEAT AND CIGARETTE BURN RESISTANCE

- 1: Coating Delaminated with both side Blisters
- 2: Coating Delaminated with single side Blisters
- 3: Brown Stain & small Blisters
- 4: Dark Brown Stain
- 5: Moderate brown Stain
- 6: Dark Brown rim
- 7: Light Brown rim
- 8: White rim & shrinkage
- 9: White rim
- 10: No change

The sample of the three woods namely Kail, Kikar and Teak were coated with epoxy diacrylate. The coated wood samples were exposed to different doses of energy viz. 100,

200, 300, 400 KGy. At 100 KGy the coating was sticky and tacky. Almost same results were observed with 200 and 300 KGy. At 400 KGy the coating surface was found cured properly. Once the proper curing of samples was achieved, the behavior of different properties has been reported. The property profile of coated teak, kail and kikar wood samples has been tabulated in Table 5, Table 6 and Table 7 respectively. The coated and cured sample was evaluated against conventional coating Polyurethane and Melamine. The results obtained are shown as:

Sl. No Properties				Coatings	1	
		ABC 1	ABC 2	ABC 3	PU	Melamine
1	Gloss	97	99	100	91	93
2	Stain resistance					
	1.NaOH	10	09	10	10	09
	2.CH ₃ COOH	10	10	8	10	10
	3.Hair dye	4	10	10	10	4
	$4.H_2O_2$	10	10	10	10	10
	5.CH ₃ COCH ₃	01	01	01	07	07
3	Resistant to dry	10	10	10	09	02
	heat					
4	Steam	10	10	10	08	01
	resistance					
5	Resistance to					
	cigarette burn					
	1.Red & white	05	04	04	05	02
	2.Ultra Mild	04	04	04	03	02
6	Pencil hardness	< 6B	< 6B	< 6B	< 6B	< 6B
7	Water	10	09	10	03	01
	absorption					

Table 5 Property profile of coated teak wood samples



Fig 15 ABC coated & uncoated teak wood samples



Fig 16 ABC coated teak wood samples for dry heat resistance



Fig 17ABC coated teak wood samples for cigarette burn resistance



Fig 18 PU coated teak wood samples for cigarette burn resistance



Fig 19 PU & Melamine coated teak wood samples for steam resistance test



Fig 20 Melamine coated teak wood samples for dry heat resistance

- 1. ABC 3 coating was found more glossy and better as compared to ABC 1 and ABC and all these samples have better gloss than conventional coating
- In case of ABC 2 & ABC 3 coatings which were exposed to acetic acid, the stain resistance showed no changes and matched to that of conventional coating while stain resistance of ABC 1 was lower
- 3. Acetone resistant of ABC 1, 2, 3 Coatings were not equivalent with conventional coatings because the surface of wood coated sample showed a tacky rim. When exposed to an acetone. This show that acetone penetrate into the coatings threw small pores which were present in the surface because of its non uniformity
- 4. The effect of NaOH, when exposed was negligible on both ABC 1, 3 and PU coatings but left a glossy mark on Melamine and ABC 2
- The stain resistance of ABC 1 & ABC 2 coating with respect to hair dye were found similar to each other and to PU coatings but different to melamine while stain resistance of ABC 1 was similar to melamine but different to PU coating and ABC 2 & 3
- 6. Thermal Resistant evaluation like cigarette burning, steam resistant and dry heat resistant of ABC coatings were better than conventional coatings because conventional coatings were not cross link as ABC coatings cross link
- 7. Water absorption of ABC coatings were better than conventional coatings
- 8. The pencil hardness were found same of ABC coatings and conventional coatings

Sl. No	Properties	Coatings				
		ABC 1	ABC 2	ABC 3	PU	Melamine
1	Gloss	104	101	57	92	88
2	Stain resistance					
	1.NaOH	09	10	10	10	10
	2.CH ₃ COOH	10	10	10	10	10
	3.Hair dye	08	08	08	05	04
	$4.H_2O_2$	10	10	10	10	10
	5.CH ₃ COCH ₃	01	01	01	07	08
3	Resistance to	10	10	10	08	07
5	dry heat	10	10	10	00	07
4	Steam	10	10	10	09	08
	resistance					
5	Resistance to					
	cigarette burn					
	1.Red & white	03	03	03	03	02
	2.Ultra mild	03	03	03	02	01
6	Pencil hardness	< 6B	< 6B	< 6B	< 6B	< 6B
7	Water	11.59	10.52	9.87	3.43	2.66
	absorption					

Table 6 Property profile of coated kail wood samples

- ABC 1, 2 coatings were proved better as compared to conventional coatings as they were more glossy, while ABC 3 was not better. In case of ABC 1, 2, 3 the gloss of ABC 1 & 2 was better than ABC 3
- Kail wood sample, coated with ABC 1, 2, 3 compositions shows no changes when they were exposed to CH₃COOH and H₂O₂. The stain resistant with respect to these matched with that of conventional coatings (PU, Melamine)
- 3. In case of Stain resistance with respect to ABC 1, 2, 3 coatings were not similar with conventional coatings because the surface of wood coated sample showed a tacky rim. This shows that acetone penetrates into the coating threw small pores which were present in the surface because of its non uniformity
- The effect of NaOH, when exposed, was negligible on ABC 2, 3, Melamine and PU coatings but left a glossy mark on ABC 1
- 5. The exposure of hair dye to different samples left a faint rim stain on ABC 1, 2, 3 but light brown stain was seen on melamine coatings while yellow stain on PU coatings due to non uniform and porous surface of these coatings
- 6. Thermal resistant evaluation like cigarette burning, steam resistant and dry heat resistant of ABC 1, 2, 3 coatings were better than conventional coatings because of cross linking
- 7. Water absorption of ABC 1, 2, 3 coatings were better than conventional coatings water absorption of ABC 1 coating was maximum among ABC1, 2, 3 samples
- 8. The pencil hardness was same as ABC coatings and conventional coatings

Sl. No	Properties	Coatings				
		ABC 1	ABC 2	ABC 3	PU	Melamine
1	Gloss	100	98	96	89	91
2	Stain resistance					
	1.NaOH	09	10	10	10	09
	2.CH ₃ COOH	10	10	10	10	10
	3.Hair dye	08	05	10	04	04
	$4.H_2O_2$	10	10	10	10	10
	5.CH ₃ COCH ₃	01	01	01	07	06
3	Resistance to	10	10	10	09	06
	dry heat					
4	Steam	10	10	10	08	07
	resistance					
5	Resistance to					
	cigarette burn					
	1.Red & white	05	05	04	03	04
	2.Ultra mild	07	05	05	04	03
6	Pencil hardness	< 6B	< 6B	5B	5B	5B
7	Water	4.70	5.73	3.92	2.16	1.56
	absorption					

Table 7 Property profile of coated kikar wood samples

- 1. ABC 1, 2, 3 coatings were proved better as compared to conventional coatings as they were more glossy in case of kikar wood. However the gloss ABC 1 was best amongst all
- 2. Kikar wood samples, coated with ABC 1, 2, 3 compositions show no changes when they were exposed to CH₃COOH, H₂O₂. The stain resistant with respect to

these chemicals found similar behavior with that of conventional coating (PU & Melamine)

- 3. Acetone resistance of ABC 1, 2, 3 coatings were not equivalent with conventional coatings because the surface of wood coated sample showed a tacky rim, when exposed to an acetone. This shows that acetone penetrates into the coating threw small pores which were present in the surface because of its non uniformity
- 4. The effect of NaOH, when exposed, was negligible on ABC 2, 3 and PU coatings but left a glossy mark on ABC 1 and melamine
- 5. The exposure of Hair dye left a faint rim stain on ABC 1, yellow stain on ABC 2 and light brown stain on PU and melamine coatings while there was no change in ABC 3. This was due to non uniform and porous surface of these coated samples
- Thermal resistant evaluation like cigarette burning, steam resistant and dry heat resistant of ABC coatings were better than conventional coatings due to the crosslinking
- Water absorption of ABC 1, 2, 3 coatings were better than conventional coatings.
 Water absorption of ABC 1 coating was maximum among all the samples
- 8. The pencil hardness of the ABC coatings were better than conventional coatings

Chapter 6

CONCLUSION

It has been observed from results and discussion that wood coated samples, ABC 1, 2 & 3, show better performance than the traditional coated woods. Some properties including resistance to the stains with respect to the hydrogen peroxide, caustic soda solution and acetic acid were better than that of conventional coatings. However, the resistance to acetone was not found satisfactorily better than that conventional coatings. In case of a Teak wood and Kail wood samples, ABC 3 produced good results while Kikar wood results were not better than ABC 1. It is also concluded that the ABC 3 the (composition

of epoxy diacrylate and Dipropylene glycol diacrylate) was also suitable for wood coating.

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GLOSSARY OF TERMS

A =1 = 4 = ()	
Acrylate(s)	An organic salt produced by the reaction of acrylic acid with an organic molecule. It is any molecule that has an acrylic radical attached to it.
Adhesive(s)	Specialty coating designed applied to a material to make it cling or stick fast to another surface (e.g., the coating applied to make pressure sensitive tape and labels).
Cationic/Cationic	A positive valence ion that is attracted to the cathode in electrolysis.
Polymerization	A cationic photoinitiator is one that decomposes
	under UV/EB radiant energy or from Bronsted Acids to form cations
	that cause polymerization to occur by an ionic mechanism
Coating(s)	A protective, adhesive, or decorative film applied in a layer to a
	Surface. The coating can be applied as either a liquid or a solid.
Crosslinking	The interconnecting of one polymer molecule with another by
	molecular bonds. Crosslinking raises the glass transition temperature and gives mechanical strength and abrasion resistance to the polymer.
Diluent(s)	Two identical molecules link together to form one molecule. An
	example is a hydrogen peroxide molecule which is two
	Hydroxyl radicals linked together.

Electron beam	Electron beams are produced by a high potential gradient between
(EB)	electrodes. When the gradient is high enough, electrons stream from
EB Radiant	the negative electrode. A positive electrode having a hole in the
Energy	middle attracts the electrons. Because an electron has mass, electrons
	that are directed toward the hole continue through as an electron
	beam.
Free Radical	A free radical is a molecular fragment having a single unpaired
/Free Radical	electron. It causes polymerization by transfer of an electron
Polymerization	From a monomer molecule. When this transfer occurs, the free
	radical and the monomer molecule become one. This new
	Molecule is a larger free radical that can then transfer an electron
	from still another monomer molecule. This process can continue in a
	chain reaction that will form a seemingly endless polymer molecule
	(one composed of many monomers).
Functional	A radical that has been attached to a monomer or oligomer molecule.
Group(s)	A functional group causes a molecule to become excited more easily
	in the presence of free radicals and radiant energy. The most
	commonly used functional groups are the acrylic, methacrylic, vinyl
	ether, and methoxyacrylic radicals.

Monomer(s)	A molecule that self-assembles into a polymer. Each monomer is another vertebra in a polymer's carbon backbone. The types of monomer that are available will define the properties of a polymer. Monomer molecules are named for their chemical structure (e.g., epoxy, polyester, urethane, s etc).
Oligomer(s)	A group of 3 to 5 monomer molecules that have been joined together to form a larger molecule. It behaves as a monomer Would, but is usually less toxic.
Oxygen Inhibition	The premature termination of a polymer molecule caused by the reaction of oxygen molecules with free radicals. When Polymerization is terminated prematurely; polymer molecules are shorter and perform differently.
Photoinitiator(s)	A compound that forms free radicals or cations when subjected to UV/EB radiant energy. A photoinitiator is sensitive to a Specific wavelength of radiant energy. Photoinitiators now become part of the polymer that is formed.
Photon(s)	According to quantum theory, a hypothetical mass less particle that is used to explain some aspects of an electromagnetic wave. In quantum theory, the electromagnetic wave and the particle are the same.
Polymer(s)	A chain of many monomer molecules linked together. This chain can be thousands to millions of monomers long. The polymer chain tangles and bonds to other polymer chains. Commonly, a polymer is

	known as a plastic.
Polymerization	The process that forms a polymer from monomers and oligomers.
Radiation	Energy (in some form) emitted from a source, propagated radially along a line-of-sight, and absorbed. However, depending on the type and source of the energy, the effects of radiation on humans are very different
Solvent	A liquid used in a paint, coating or ink to dissolve or disperse film- forming constituents and to adjust viscosity. It evaporates during drying/curing and does not become part of the dried film.
Substrate	The surface to which a coating is applied.
Volatile Organic	Any compound of carbon, excluding carbon monoxide, carbon
Compounds	dioxide, carbonic acid, metallic carbides or carbonates, and
(VOC)	Ammonium carbonate, which participates in atmospheric photochemical reactions. This includes any such organic compound other than those that the Administrator of EPA has determined to have negligible photochemical reactivity.

Ultraviolet (UV)	That portion of the electromagnetic spectrum between 180 nm and
UV Radiant	400 nm. This can be broken into near-UV (315 nm- 400 nm) and far-
Energy	UV or actinic (180 nm - 315 nm). It can also be broken into UV-A (
	315 nm - 400 nm), UV-B (290nm -315
	nm), and UV-C (220nm - 290 nm). Near-UV, UV-a, and UV-B are
	used for curing UV materials. Sunburn is caused by UV-A
	And UV-B. Actinic UV or UV-C is used for sterilization.