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## PROCEEDINGS

# WORKSHOP ON SURFACE FINISHING BY RADIATION CURING TECHNOLOGY

8 - 9 September, 1993,  
BANGI

# **RADIATION CURING FOR BETTER FINISHING**

Organized by

**Unit Tenaga Nuklear (UTN)  
Japan International Co-operation Agency (JICA)  
and  
International Atomic Energy Agency (IAEA)**

U.V. VARNISH

# **WORKSHOP BANGI, 8-9 SEPT. 1993**

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**National Executive Management Workshop on Surface Finishing by Radiation  
Curing Technology.**

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**NATIONAL EXECUTIVE MANAGEMENT WORKSHOP ON SURFACE  
FINISHING  
BY RADIATION CURING TECHNOLOGY  
( 8th & 9th September 1993, Bangi )**

**OPENING ADDRESS BY DIRECTOR GENERAL OF UTN**

Mr. Chairman, Distinguished Speakers, Ladies and Gentlemen.

It is a great pleasure for me to be given a chance to inaugurate this workshop today. On behalf of the organizing committee, I would like to express my warm welcome to all of you, the distinguished speakers and the participants. Some of you already attended the seminar yesterday on the same subject. I believe to some extent you are already familiar with the concepts and jargons of surface finishing by radiation curing. Therefore from the workshop, I am sure you can benefit even more.

The workshop has been arranged in such a way that the more details of this technology are covered. We feel that certain areas such as the chemical and hardware aspects of the technology need to be covered in separate topics. Selection and preparation of materials to be coated is also another important areas and need to covered separately as well. We also include some demonstrations on how radiation techniques are carried out in surface finishing. The participants will also be exposed to our R&D activities in radiation curing of surface finishing. The works are mainly on two areas i.e. formulation of commercial oligomers and developments of new resins based on palm oil and natural rubber. These new developments will be discussed in more detail in this workshop.

The workshop is being conducted here in our premises so that you have an opportunity to see for yourselves our activities and facilities, not only in the field of radiation curing but also other areas of utilisations. Radiation curing is only one area of many other areas that mankind can benefit from the peaceful use of nuclear techniques.

Ladies and gentlemen,

Malaysia is fast becoming an industrialising country based on manufacturing sector. It is the intention of the government to see local industries to be adaptive with new technology. In order to be competitive, they need to be innovative, and ready to adopt new technological changes in the productive process and other downstream activities.

For Malaysia, the wood based industry is in a good position to adopt this relatively new technology. The industry contributes very significantly to the nation's revenue, last year alone, its export values were more than RM10 billion-second largest export earner among primary commodities after petroleum. Sawntimber contributed 53% to the total production of wood based products, while plywood, hard board and particle board accounted for 47%.

At present, downstream processing of wood products comprise mainly the production of plywood, veneer and mouldings. Therefore, the potential of the industry is still not fully realised and the progress into higher value added products is still slow. Nevertheless, if we look at furniture industry, in 1991 it registered a tremendous growth in terms of exports earning, even though in terms of the overall industry value, is still small. The uptrend is expected to be maintained in the coming years.

It is strongly felt that the radiation techniques could further improve the situation. The entry of the wood based industry into further value added manufactures will provide greater diversification opportunities.

At present, there are already a number of local wood companies using the UV technique for curing of coating. But the technique is more widely used by printing companies and electronic factories. So far EB accelerator is yet to be in commercial use in the country.

UTN by its virtues of being a national nuclear and radiation technology research centre, can play a major role, together with other government institutions and private sector in promoting and facilitating the acceptability of this technology by local industry. In this regard, UTN is always looking forward to collaborative works with any other organizations, be it from public sector or private sector.

Ladies and gentleman,

The organizers are indeed very pleased and grateful at the response of all concerned without whom this workshop will undoubtedly be a failure. The government research institute will definitely benefit from the interest and the participation of the private sector. With that note, I officially declare this workshop open.

Thank you.

#1

# AN INTRODUCTION TO RADIATION CURING





## **An Introduction to Radiation Curing**

**Takashi Sasaki**

**Takasaki Radiation Chemistry Research Establishment,  
Japan Atomic Energy Research Establishment**

**Workshop**

**on**

**Surface Finishing by Radiation Curing Technology**

**8 -9 September 1993**

**Unit Tenaga Nuclear, Bangi**

**Selangor, Malaysia**

## AN INTRODUCTION TO RADIATION CURING

T. Sasaki

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Japan Atomic Energy Research Institute

### Introduction

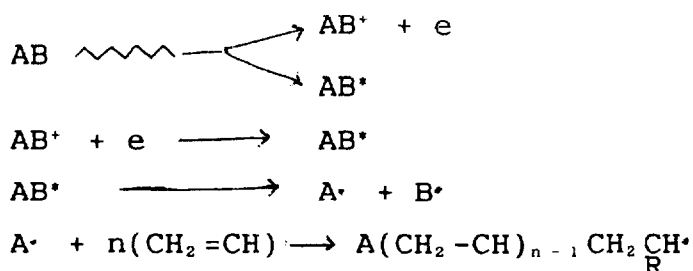
Radiation curing essentially involves the application of a thin coating of a viscous prepolymer-monomer mixture onto a substrate, followed by passage under a radiation source to solidify the coating. The original meaning of "cure" or "curing", commonly used in the conventional systems, is merely a physical change from a liquid state to a solid state. However, the change in the radiation systems is based on chemical reactions, normally radical (co-)polymerization between double bonds in the prepolymer (oligomer) and monomer(s) as schematically illustrated in Fig. 1. The reaction is very fast due to high concentration of radicals yielded in the mixture under the beam or UV light.

Besides the free radical chemistry, there are other radiation curing systems using initiators which propagate cationic polymerization. Major advantages of radiation curing systems are summarized in Table 1.

### Initiation of Radical Polymerization

#### Initiation by Ionizing Radiation

Ionizing radiations transfer their energy to matter through electrostatic interaction of fast-moving electrons with the orbital electrons of the irradiated substance. Mainly ions and excited molecules are initially produced through primary and (repeating) secondary ionization or excitation. These species are rapidly converted into free radicals which are, in most cases, the active chemical species to initiate polymerization:



It should be noted that the rate of formation of radicals is temperature-independent (as in photochemical initiation). Thus, the polymerization can readily take place at room temperature or even at lower temperatures.

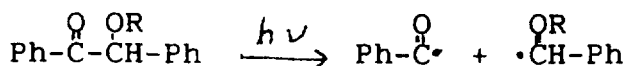
Although most of early studies on radiation polymerizations confirmed that they proceed by a free-radical mechanism, several evidences demonstrated that ionic polymerizations could be initiated by irradiation at such conditions as 'super dry' or solid states of specific monomers.

#### Photo-Initiation

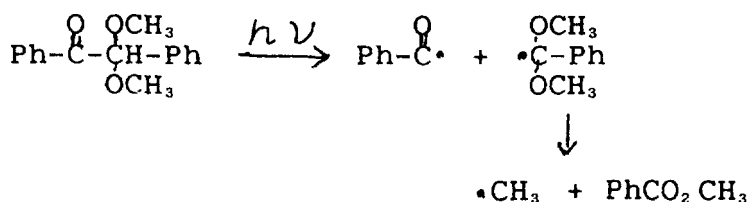
The energy range of UV radiation lies between 5 to 100 eV and is almost equivalent with that of the ionization potentials of organic matter. Therefore, UV radiation does not cause direct ionization of organic substrates. Necessarily, polymerization are achieved with photo-sensitive molecules (photoinitiator) which absorb UV energy efficiently to form excited states and then photolize to generate free radicals. There are two general classes of photoinitiators:

##### 1) Direct photofragmentation.

Alkyl ethers of benzoin.



(further rearrangement decomposition)



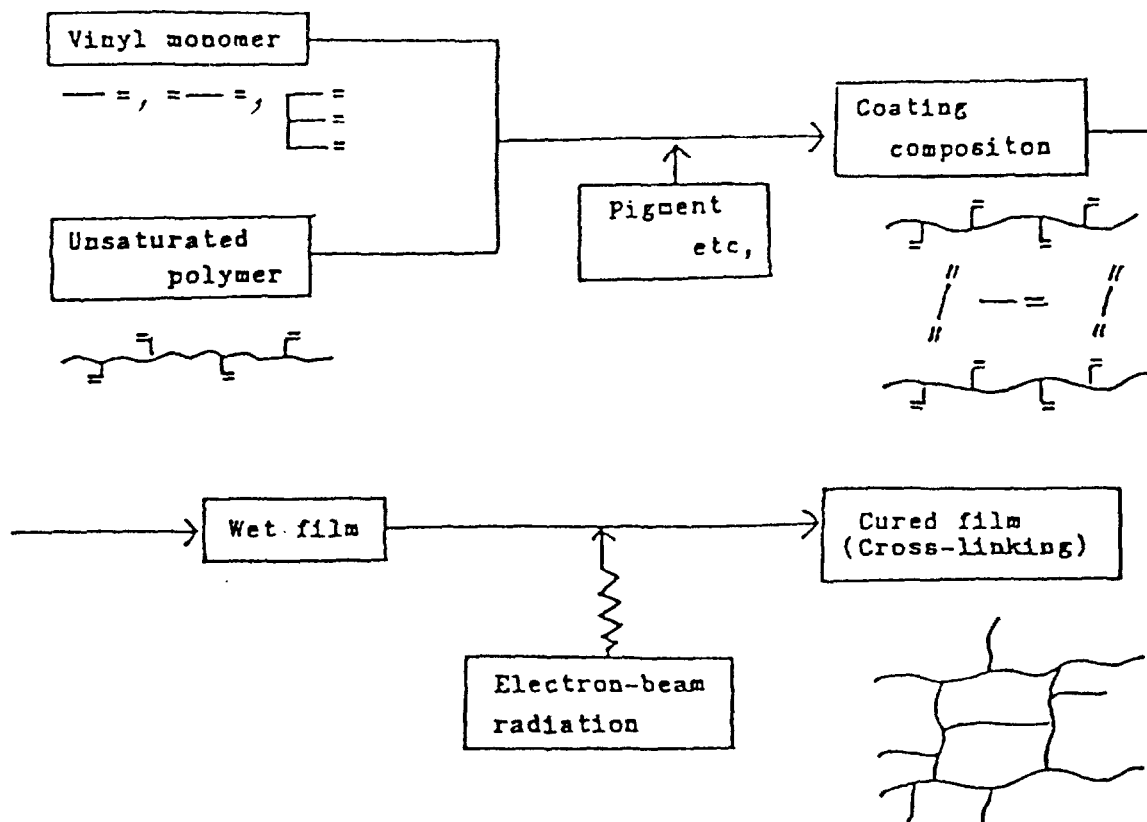


Fig. 1 Schematic diagram of electron beam curing

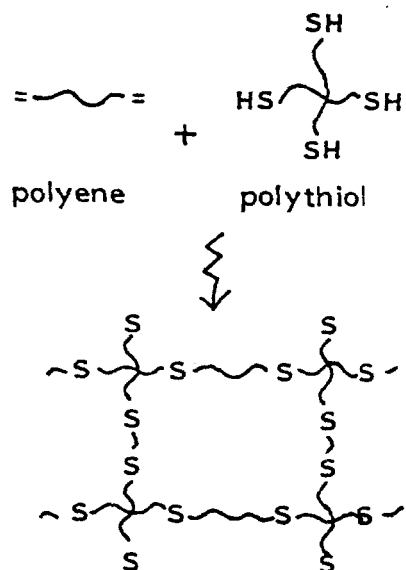
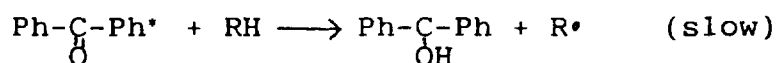


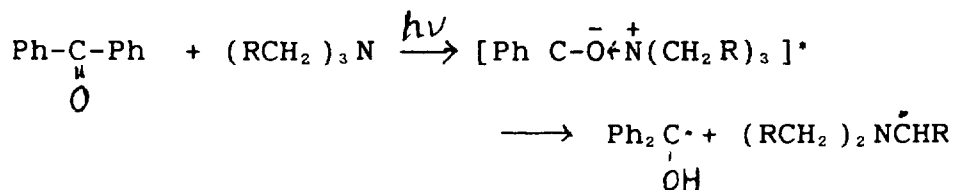
Fig. 2 Crosslinked network formation in polyene/thiol system

## 2) Proton/electron transfer

## a) Benzophenone



## b) Benzophenone + tert-amine



Since a tert-amine compound also forms a complex with a peroxide radical to generate a reactive free radical, polymerization of this system is not so much affected by the presence of oxygen.

## Factors in Free-Radical Polymerization

## Effect of Oxygen

Free radicals are generally much more reactive with oxygen than monomers to form peroxide radicals or further hydroperoxides which are very stable and inhibits radical polymerization. Therefore, any radical polymerization process (including catalytic systems in principle) should be performed under an inert (oxygen free) condition.

## Influence of Dose Rate

The rate of polymerization,  $R_p$ , is dependent upon the concentration of free radicals,  $[R^{\bullet}]$ , and the concentration of the reactants,  $[M]$ . Assuming a steady state of free radicals and bi-molecular termination of polymerization, the following relationship can be derived:

$$R_p \propto [R^{\bullet}][M] \propto [I]^{1/2}[M]$$

where,  $[I]$  is the intensity of electron beam or the dose rate.

This relationship, well known as the square root relationship, is not always valid for practical cases, which indicates the assumptions are too much simplified. It should also be noted

that the rate of gel formation is not necessarily proportional to that of polymerization.

The situation being such, the dependence of dose-to-cure (Dc) on dose rate is generally expressed as:

$$Dc \propto [I]^\alpha$$

where  $\alpha$  lies between 0 and 0.5. Anyhow, it can generally be said that a simple linear relationship does not exist between the line speed and the dose rate.

## Radiation-Curable Materials

### Prepolymers

Radiation curable prepolymers generally are low- to medium-molecular weight mono- or multi-functional unsaturated materials, and can be categorized as:

- 1) Unsaturated polyesters
- 2) Acrylated prepolymers
  - Unsaturated acrylics
  - Urethane acrylates
  - Polyester acrylates
  - Polyether acrylates
  - Epoxy acrylates
- 3) Polyene/thiol systems

They provide the primary properties of the cured film such as high abrasion resistance, high tensile strength, elongation, good solvent resistance, and acceptable levels of hardness and flexibility.

Unsaturated polyesters are well known as one group of thermosetting resins and have been used for fiber reinforced plastics (FRP) and coatings. They are normally being used as mixtures with styrene, and comparatively cheap in price. UPE-styrene mixtures can be used for wood coatings, but the cure rate is rather slow.

Acrylated prepolymers can be cured much faster than UPE-styrene mixtures, but are generally costly. Structures and

characteristics of these prepolymers are shown in Table 2.

Polyene/thiol systems are very unique compared with other ones and have little effect of oxygen inhibition on the cure rate, but have troubles in odors. The reaction scheme of the systems is illustrated in Fig. 2.

### Monomers

Prepolymers, being generally highly viscous or almost solid, are necessary to be diluted with monomer(s) to have suitable working viscosities. A monomer will influence cure rate, mechanical and physical properties, adhesion, residual odor, and also toxicological properties of the solutions or cured films. Many monomers are available as listed in Table 3.

### Cationic Initiation

As mentioned before, polymerization can be initiated by a cationic mechanism under a certain limited condition which is practically impossible for curing of coatings.

Since the types of prepolymers which can be cured by radical polymerizations are limited, efforts have been done to develop new type of photo-initiators to enable to cure epoxy resins by cationic mechanism. Various kinds of aromatic onium salts of Lewis acid have been developed for this purpose. These salts are thermally stable, but photolyze to strong Lewis acids which initiate cationic polymerization.

These onium salts are also usable for EB curing, although the decomposition mechanism of the is somewhat different from that in the UV irradiation. Major advantages of the cationic systems are that the dose rate (Fig. 3) and the presence of oxygen (Table 4) do not effect the cure rate.

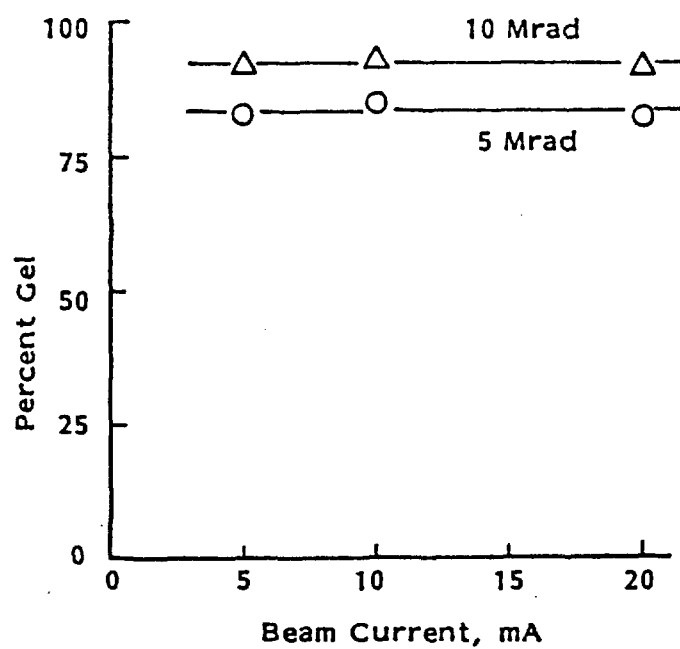


Fig. 3 Effect of dose rate on gel formation in cationic system

Resin: Polybutadiene modified epoxy (EPB-27)

Onium salt:  $\text{Ph}_2\text{IPF}_6$  (10% in PGE)



Table 1 Advantages of Radiation Curing Systems

Hardening at Ambient Temperature
Energy-saving
Applicable to Heat Labile Substrates
Rapid Curing --- High Speed Line
100 % Solid System (Solvent-Free)
Less Materials and Non-polluting
Non-Catalyst Systems (EB)
Long Potlife of Resins
Better Weather Durability of Products
Rapid Start-up and Shut-down of Power Source
Less Plant Space

Table 2 Acrylic Prepolymers

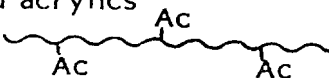

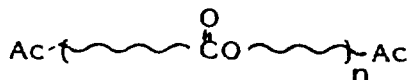
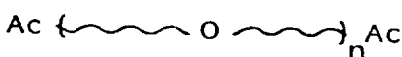
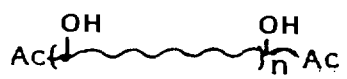
Unsaturated acrylics		high weather durability chemical stain resistance
Urethane acrylate		flexibility abrasion resistance
Polyester acrylate		hardness stain resistance
Polyether acrylate		flexibility elongation
Epoxy acrylate		good adhesion chemical stain resistance

Table 3 Examples of monomers

a) Monofunctional

Styrene	$\text{CH}_2=\text{CH}-\text{C}_6\text{H}_5$	145
Vinyl toluene	$\text{CH}_2=\text{CH}-\text{C}_6\text{H}_4-\text{CH}_3$	o, 52/9 m, 51/3 p, 66/18
Butyl acrylate	$\text{CH}_2=\text{CHCOOC}_4\text{H}_9$	n, 147 i, 138
2-Ethylhexyl acrylate	$\text{CH}_2=\text{CHCOOCH}_2\text{CH}(\text{C}_2\text{H}_5)\text{C}_4\text{H}_9$	130/50
Isodecyl acrylate	$\text{CH}_2=\text{CHCOO}(\text{CH}_2)_7\text{CH}(\text{CH}_3)_2$	
2-Hydroxyethyl acrylate	$\text{CH}_2=\text{CHCOO}(\text{CH}_2)_2\text{OH}$	75/5
2-Hydroxypropyl acrylate	$\text{CH}_2=\text{CHCOOCH}_2\text{CH}(\text{OH})\text{CH}_3$	193
Cyclohexyl acrylate	$\text{CH}_2=\text{CHCOO}-\text{C}_6\text{H}_{11}$	
N,N'-Dimethylaminoethyl acrylate	$\text{CH}_2=\text{CHCOOCH}_2\text{CH}_2\text{N}(\text{CH}_3)_2$	60/10
N-Vinyl pyrrolidone	$\text{CH}_2=\text{CH}-\text{N}-\text{C}_4\text{H}_7\text{=O}$	123/50
Carbitol acrylate	$\text{CH}_2=\text{CHCOO}(\text{CH}_2)_2\text{O}(\text{CH}_2)_2\text{OC}_2\text{H}_5$	
Phenoxyethyl acrylate	$\text{CH}_2=\text{CHCOOC}_2\text{H}_4-\text{O}-\text{C}_6\text{H}_5$	
Tetrahydrofurfuryl acrylate	$\text{CH}_2=\text{CHCOOCH}_2-\text{C}_4\text{H}_7\text{O}$	
Isobornyl acrylate	$\text{CH}_2=\text{CHCOO}-\text{C}_{10}\text{H}_{17}$	
2-(N-Methylcarbamoyl) acrylate	$\text{CH}_2=\text{CHCOOCH}_2\text{CH}_2\text{OCONHCH}_3$	
3-Butoxy-2-hydroxy-propyl acrylate	$\text{CH}_2=\text{CHCOOCH}_2\text{CH}(\text{OH})\text{CH}_2\text{OC}_4\text{H}_9$	

b) Multifunctional Monomers

Compound	Structure
1,4-Butanediol diacrylate	$\text{CH}_2=\text{CHCOO}(\text{CH}_2)_4\text{OCOCH}=\text{CH}_2$
1,6-Hexandiol diacrylate	$\text{CH}_2=\text{CHCOO}(\text{CH}_2)_6\text{OCOCH}=\text{CH}_2$
Neopentyl glycol diacrylate	$\text{CH}_2=\text{CHCOOCH}_2\text{C}(\text{CH}_3)_2\text{CH}_2\text{OCOCH}=\text{CH}_2$
Diethylene glycol diacrylate	$\text{CH}_2=\text{CHCOO}(\text{CH}_2\text{CH}_2\text{O})_2\text{COCH}=\text{CH}_2$
Polyethylene glycol diacrylate	$\text{CH}_2=\text{CHCOO}(\text{CH}_2\text{CH}_2\text{O})_n\text{COCH}=\text{CH}_2$
Pentaerythritol diacrylate	$(\text{CH}_2=\text{CHCOOCH}_2)_2\text{C}(\text{CH}_2\text{OH})_2$
Trimethylolpropane triacrylate	$(\text{CH}_2=\text{CHCOOCH}_2)_3\text{CC}_2\text{H}_5$
Pentaerythritol triacrylate	$(\text{CH}_2=\text{CHCOOCH}_2)_3\text{CCH}_2\text{OH}$
Triallyl cyanurate	$  \begin{array}{c}  \text{OCH}_2\text{CH}=\text{CH}_2 \\    \\  \text{N}=\text{C} \quad \text{N} \\  \diagdown \quad \diagup \\  \text{CH}_2=\text{CHCH}_2\text{O}-\text{C} \quad \text{C}-\text{OCH}_2\text{CH}=\text{CH}_2 \\  \diagup \quad \diagdown \\  \text{N} \quad \text{N}  \end{array}  $
Trimethylolpropane triallyl ether	$  \begin{array}{c}  \text{CH}_2\text{OCH}_2\text{CH}=\text{CH}_2 \\    \\  \text{CH}_3\text{CH}_2-\text{C}-\text{CH}_2\text{OCH}_2\text{CH}=\text{CH}_2 \\    \\  \text{CH}_2\text{OCH}_2\text{CH}=\text{CH}_2  \end{array}  $
Pentaerythritol tetraacrylate	$\text{CH}_2=\text{CHCOOCH}_2\text{C}(\text{CH}_2\text{OCOCH}=\text{CH}_2)_3$

Table 4. Effect of oxygen in cationic system

Resin	Atmos- phere	Percent Gel (Tack)			
		2 Mrad	4 Mrad	6 Mrad	8 Mrad
14	Film	85	89	91	91(XX)
	Air	76	85	87	90(X)
27-1	Film	31	69	76(O)	81(O)
	Air	20	61	77(O)	78(⊙)
27-2	Film	56	77	84(Δ)	88(⊙)
	Air	41	72	84(O)	90(⊙)

#2

**RADIATION SOURCES:  
EB & UV CURING  
MACHINE**



MY9700871

**Radiation Sources  
EB and UV Curing Machines**

**Takashi Sasaki**  
**Takasaki Radiation Chemistry Research Establishment,**  
**Japan Atomic Energy Research Establishment**

**Workshop**  
**on**  
**Surface Finishing by Radiation Curing Technology**  
**8 -9 September 1993**  
**Unit Tenaga Nuclear, Bangi**  
**Selangor, Malaysia**

## ELECTRON BEAM PROCESSORS FOR RADIATION CURING APPLICATIONS

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### INTRODUCTION

The electron beam (EB) curing system acquired an industrial reality in late 1960's when so-called low energy EB accelerators with industrial level performances became available. The process involves an instantaneous change in state of a liquid prepolymer-monomer mixture to a solid thin coating by free radical polymerization with crosslinked structure. Oxygen, being very reactive with growing radicals to form stable chemical species, inhibits the radical polymerization, which leads the system to be processed in an inert atmosphere using either (liquid) nitrogen or combustion gas.

Major advantages of the EB curing processes are as follows: a solvent-free system, less energy consumptive, a much higher production rate, and a processing ability at ambient temperature. The equipment is very compact with resultant space savings and able to be easily controlled. Most of the commercialized products using this technology are also of improved qualities and the application areas of the technology are expanding.

This paper describes electron beam processors and related technologies for curing applications to facilitate those industrial persons who are trying to understand and evaluate the applicability and benefits of radiation curing to their products.

## ENERGY DEPOSITION

The electron beam curing process is the result of a transfer of energy from the accelerated electrons as they penetrate the material which is being irradiated. The penetrating capability of electrons is shown in Fig. 1. As can be seen, the higher the kinetic energy of the electron beam, the greater the penetration depth will be into the material. Fig. 2 shows energy deposition profiles as a function of the accelerating terminal voltage. It can be understood that the majority of coatings are curable with irradiation of electrons accelerated up to 300 keV. And, those accelerators in the range of 150 - 300 kV are called as low energy accelerators.

The measure of energy absorbed in a material is referred to as dose, of which unit is defined as rad or Gray (Gy) in SI unit.

$$1 \text{ rad} = 100 \text{ erg/g}, \quad 1 \text{ Gy} = 1 \text{ J/kg}$$

$$100 \text{ rad} = 1 \text{ Gy}$$

$$1 \text{ Mrad} = 10 \text{ kGy} = 2.4 \text{ cal/g}$$

Although the penetration depth of accelerated electrons is small, through collision to matter they generate X-rays which can penetrate far into materials. Therefore, the electron accelerator and its associated product treatment station must be shielded with either an exterior vault (vault-shielded or integrated structural housing of the unit (self-shielded). Accelerators operating in the energy range below 500 kV can be equipped with self-shielded models.

## ELECTRON BEAM ACCELERATOR

The working principle of an EB accelerator is similar to the Braun tube for a television set, an electron gun for an electron microscope, or a large radio-valve. That is, electrons are generated by heating a cathode material (filament) and subsequently accelerated to a desired energy electrically in such

a high vacuum as  $10^{-6}$  Torr (Fig. 3). The accelerated electrons are then pass through a thin metal window, which is made from either titanium or aluminium foil, and must be cooled to prevent its melting.

Accelerators on the market can be divided in two types; scanned beam and non-scanned beam types, as shown in Fig. 4.

In the scanning types (Fig 4a), the electrons are emitted by a spiral cathode with a diameter of about 5 mm. and the beam spot is scanned across the material to be irradiated with a frequency of around 200 Hz. Since an accelerator of this type needs a scanhorn with a height of about 1 to 2 times the width of the material, it is often positioned horizontally.

As an example of scanning type, the specifications of the accelerator which has been installed at CAIR-BATAN as a pilot-pilot-demonstration line for wood coating since 1984 are shown below:

Type: Insulated gas sealed transformer  
 EB Energy: 300 keV max.  
 EB Current: 50 mA max.  
 Scanning width: 120 cm  
 Scan frequency: 200 Hz  
 Dose uniformity:  $\pm 5$  % over a full scanning width  
 X-ray shielding: Self-shielded, shutter-type  
 X-ray leakage: below 0.2 mrem/hr  
 Conveyer speed: 2.5 - 25 m/min  
 Inert gas: Liquid nitrogen, 100 Nm<sup>3</sup>/hr max

Non-scanning type machines as shown in Fig. 4 are also called as linear-cathode type, in which the electrons are emitted by one or more linear filaments, arranged across (Fig. 4b) or parallel (Fig. 4c) to the production line. Having no scanhorn, a machine of this type can be designed more compact than a scanning-type one. The acceleration voltage is limited upto 300 kV. Typical



specification of these systems shown elsewhere are:

Voltage	150 - 300 kV
Width	15 to 250 cm
Dose	1 Mrad at 1500 m/min max
Uniformity	$\pm 5\%$
Vacuum	$5 \times 10^{-7}$ torr
Oxygen Exclusion	5 ppm
Expected Filament Life	10,000 hrs

Maintenance schedules

Air Filter	Every 2 weeks
Foil	4 months
Water Filter	2 months
SF <sub>6</sub> Pressure	2 months
Vac Pump Oil	9 months

There is another type of machine, where electrons are generated through secondary emission from a metal target (cathode) struck by high energy positive ions, as shown in Fig. 5. This type of machine can be operated at relatively low vacuum ( $10^{-2}$  Torr). The life of a secondary emitter can be considerably longer than a thermal emitter. Typical specifications of this type of machines are:

Voltage	150 to 250 kV
Current	.150 to 1.5 mA/cm <sup>2</sup> continuous 5 A/cm <sup>2</sup> pulsed
Window Areas	10 to 2000 cm <sup>2</sup>
Beam Uniformity	$\pm 10\%$
Life	>20,000 hrs continuous 10 <sup>10</sup> shots pulsed
Efficiency	60 to 80% at 175 kV
Operating Pressure	$10^{-2}$ Torr He

Quite recently, a German company which have three different EB units for its coating lines reported their experience on using them. The characteristics of the accelerators installed in the company are shown in Table 1. And, Table 2 shows maintenance time of the accelerators. It should be noted that the report concluded as follows:

"I (W. Karmann) wish to point out that the electron beam processors are accepted components of our coating line, which are employed particularly where high precision of production is required. The reliability is comparable to the other components of the lines."

#### IRRADIATION ENGINEERING

##### Automatic Control

Since an industrial electron accelerator is installed as an element of a production line, it should perform its functions without requiring the special attention of an operator. For this purpose, EB processors with automatic control system are available. Fig. 6 shows an example of such a system. By setting the required dose  $D$ , the comparator  $C$  determines the ratio of  $D$  of and signal  $T$  from the tachometer, which is proportional to the line speed  $v$ . The comparator then instructs the grid control  $G$  of the electron accelerator to adjust the beam current  $I$  according to the following relationship:

$$D = \frac{k \times I}{v}$$

where  $k$  is the yield factor of the processor ( $\text{Mrad m min}^{-1} \text{ mA}^{-1}$ ).

##### 3-Dimensional Objects

It is generally considered that electron beams travel in straight lines and therefore are most suitable for processing flat objects. When they pass through the foil and gas layer, however, scattering of electrons occurs as well as energy loss.

The lower the energy of electrons, the greater the scattering. For example, the mean scattering half-angles of 200 keV electrons for various configs are shown below:

Configuration	$\theta_{1/2}$ (degree)
1/2 mil Ti window	21
1 mil Ti window	30
1/2 mil Ti window + 2.5 cm N <sub>2</sub>	37
1/2 mil Ti window + 5.0 cm N <sub>2</sub>	43

It can be seen that the mean scattering half-angle of electrons 5 cm below the window is more than twice of that at its back side. This means an increase in the front surface dose in spite of a decrease in the penetration depth of the electrons into the product. Fig. 7 shows the effect of the air gap on the gel content of a coating. It can be clearly seen that the coating can be most effectively cured at a very distant place, not near the window. Moreover, it seems that the low energy electrons may possibly cure coatings on shaped materials. In fact, it has been shown that it is possible to cure the coating on car wheel rims.

Fig. 8 shows a schematic diagram of EB curing of coatings on car wheel rims which was installed in West Germany. The pre-coated wheels with an water-based primer are stoved followed by spray application of and EB curable metallic paint to the outside of the rims. The coated articles are then admitted to the circular curing chamber which rotates to permit the coating be irradiated and cured under the accelerator.

Other potential processes for shaped materials are shown in Fig. 9 and 10.

#### Inerting System

Because the radical initiated polymerisation reactions are inhibited by the presence of oxygen, the irradiation zone must be maintained as an oxygen-free (inert) atmosphere, typically

with oxygen levels from less than 50 ppm to 500 ppm. This can be achieved by purging with inert gas usually from either liquid nitrogen or a gas generator. The prices for liquid nitrogen vary considerably from one geographical location to another, or with the amount to be used. The consumption of inert gas on a particular line depends on a number of factors such as the line speed, line width, thickness of the products, design of the curing chamber, etc. Anyhow, the cost for inerting is pointed as one of the major disadvantages of the EB curing technology.

A inert gas recycling system has been developed by a Japanese EB machine manufacture, with a consideration of the temperature rise in the irradiation zone, in order to minimize the consumption of costful liquid nitrogen.

Table 3 shows the cost estimation for the inerting system with or without nitrogen recycling system, which is made on the following assumptions:

Acceleration voltage	200 kV
Beam current	300 mA
Max. temperature rise	25 °C
Oxygen concentration	<100 ppm
Price of liq. nitrogen	40 yen/Nm <sup>3</sup>
Electric power for recycling	5.5 kW
Electricity cost	25 yen/kWh
Annual operation time	32,000 (16 x 200) hrs
Fresh nitrogen	100 Nm <sup>3</sup> /hr
Recycled nitrogen	150 Nm <sup>3</sup> /hr

It can be clearly seen that there is a lot of cost saving when the recycling system is used.

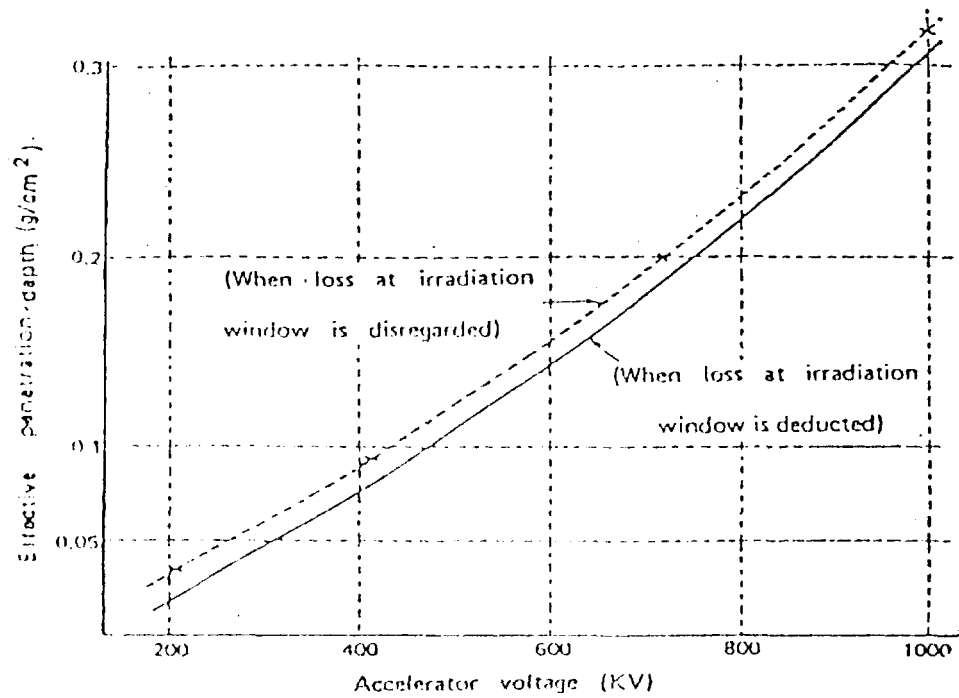


Fig. 1 Effective penetration depth

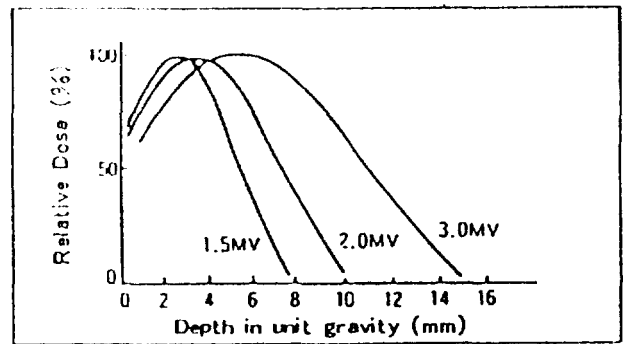
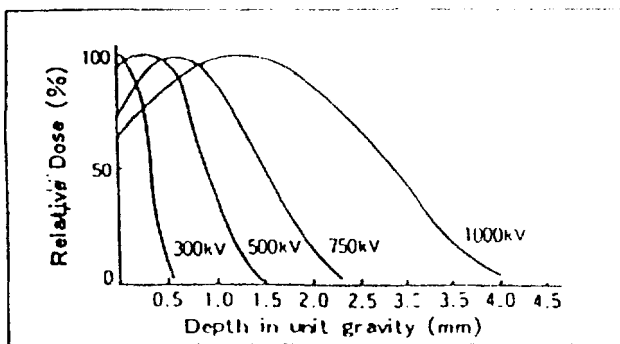


Fig 2. Energy deposition profiles.

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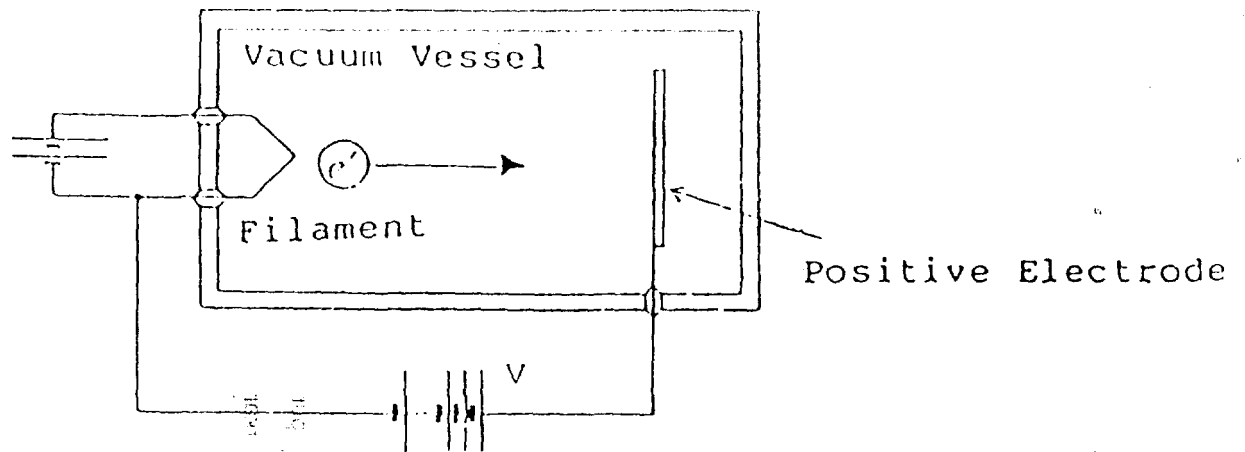


Fig 3. Principle of electron acceleration.

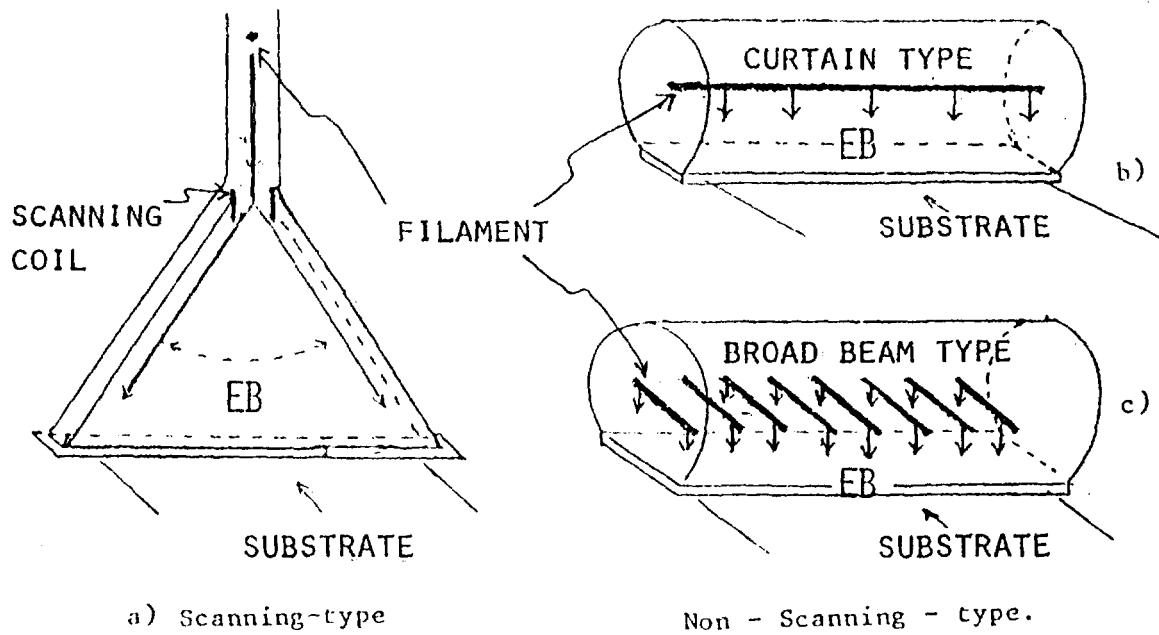


Fig. 4 Schematics of low-energy accelerators

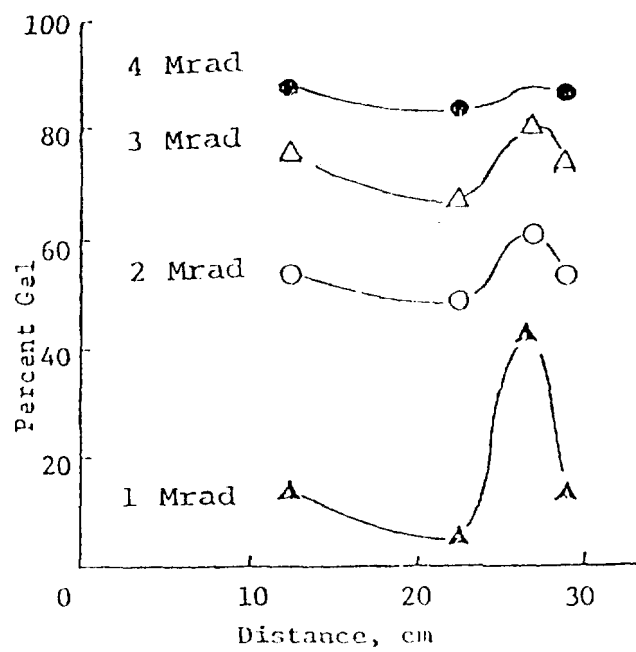


Fig. 7 Effect of distance (air-gap) on curing of an unsaturated polyester resin (300 kV scanner type)

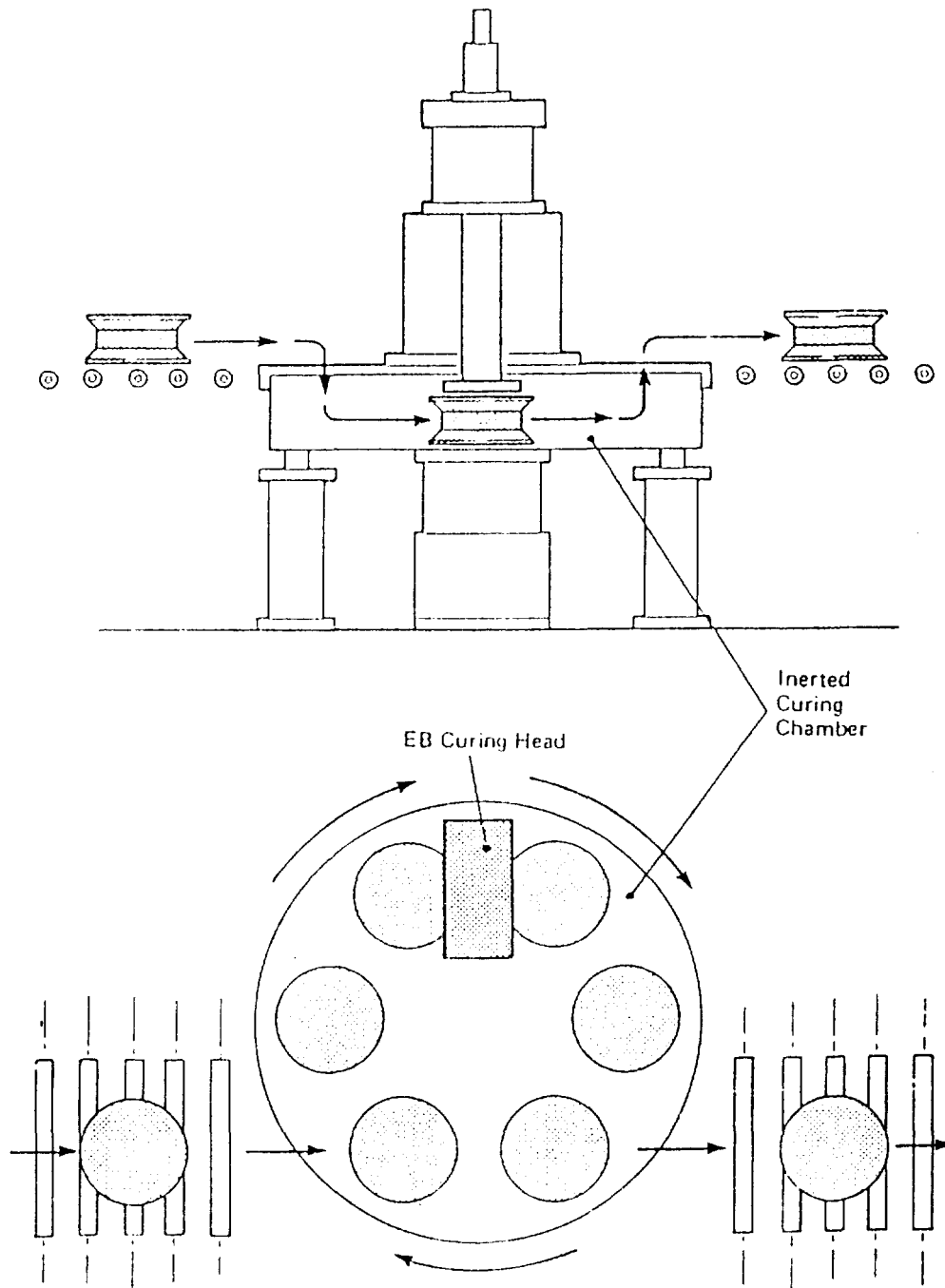


Fig. 8 Schematic diagram of EB curing of coatings on car wheels.



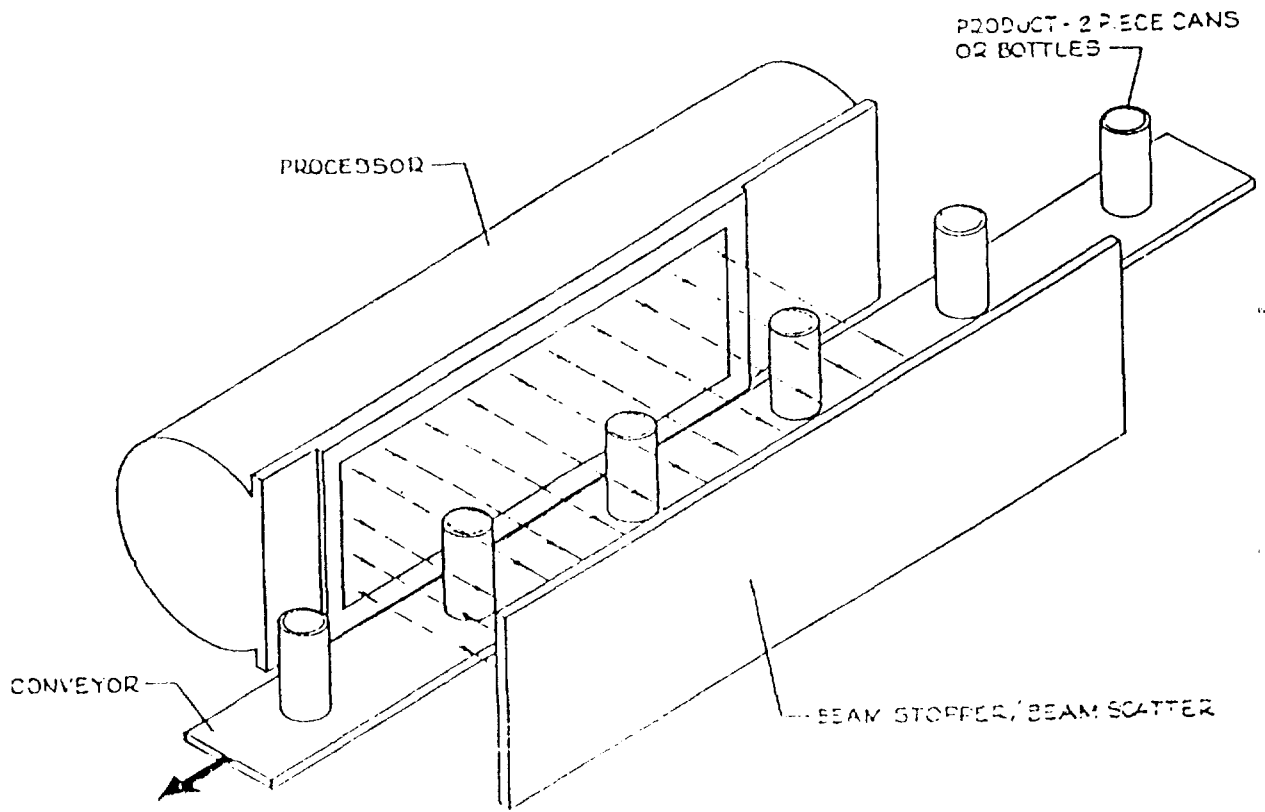


Fig. 9 Irradiation of two-piece cans.

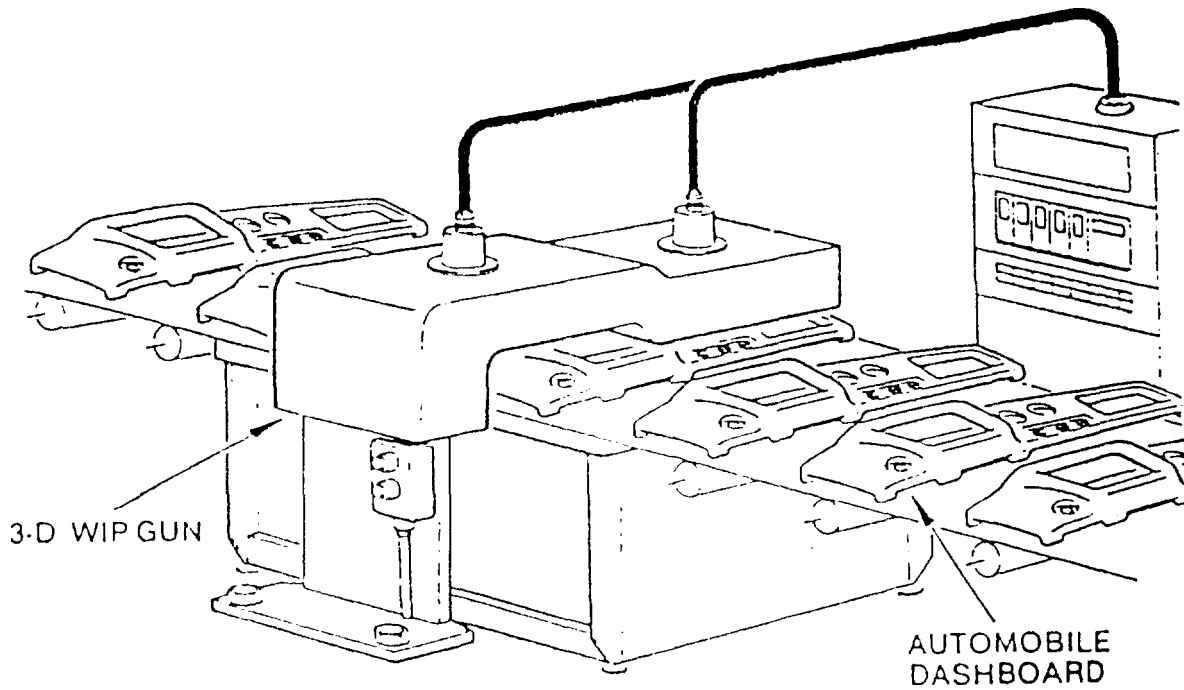


Fig. 10 Irradiation of automobile parts

Table 1 Characteristics of Accelerators at Beiersdorf AG.

Made by	:	High Voltage Engineering Corp.	Polymer-Physik GmbH	Energy Sciences Inc.
Type	:	Scanning	Scanning	Linear Cathode
Cathode	:	Spiral	Spiral	Transvers.Wire
Beam current controlled by	:	Temperature	Temperature	Grid-Voltage
Area weight of window + air gap	:	242 g/m <sup>2</sup>	110 g/m <sup>2</sup>	100 g/m <sup>2</sup>
Support	:	none	drilled plate	lamellae
Beamwidth	:	1.22 m	1.35 m	1.35 m
Max. beam current	:	0.4 mA/cm	1 mA/cm	3 mA/cm
Rise time to maximum	:	45 sec	20 sec	15 sec
In operation since	:	1980	1983	1986
Hours of operation	:	11,000	18,000	8,500

Table 2 Maintenance time of the accelerators shown in Table 1

Made by	:	High Voltage Engineering Corp.	Polymer-Physik GmbH	Energy Sciences Inc.
Lifetime of cathode	:	>11,000 h	400 h	ca.3,000 h
Average time spent *	:	ca.2 days	1 h	24 h
Lifetime of window	:	250 h*	2,000 h*	>1,000 h*
Average time spent *	:	24 h	3 h	24 h

## Explanations:

- \* usually broken by thermal stresses in the metal sealing ring
- \* usually changed preventively
- \* including reconditioning

Table 3 Cost estimation of N<sub>2</sub> recycling system

Nitrogen Recycling	YES	NO
Annual Nitrogen Consumption	320,000 Nm <sup>3</sup>	800,000 Nm <sup>3</sup>
Annual Nitrogen Cost ( 1000 yen )	12,800	32,000
Electricity for Nitrogen Recycling	17,600 kWh	0
Electricity Cost, ( 1000 yen )	440	0
Total Cost, ( 1000 yen )	13,240	32,000

## U.V. EQUIPMENT

UV equipment suitable for curing is made up from the following components:

UV source

Reflectors

Ancillary Equipment

## UV Source

## Mercury Vapor Lamps

The mercury vapor lamp is normally a sealed transparent quartz tube into which has been introduced. The length of lamps vary up to 120 cm. Electrodes made of tungsten are located at either end. A typical lamp construction is illustrated in Fig. 1.

Low pressure mercury vapor lamps: This type of lamps operate at pressures in  $10^{-2}$  to  $10^{-3}$  Torr region, and at relatively low temperatures ( $40^{\circ}\text{C}$ ). A small amount of argon gas is also introduced into the tube. The dominant wavelength emitted are 185 and 253.7 nm, the longer of the two being the more common.

Medium pressure mercury vapor lamps: These lamps are the most widely used for curing processes. The tube is constructed of transparent quartz and runs between 40 and 120 W/cm linear lamp with pressures of  $10^2$  Torr. The surface temperature of the lamp being  $700^{\circ}\text{C}$ , it needs to be cooled to control the temperature, which helps to maintain a steady spectral output. The efficient removal of any ozone produced should be ensured with appropriate extraction equipment. At the higher pressures and temperatures, a much wider spectrum output is produced than that of a low pressure lamp. Thus, the 253.7 nm line is relatively insignificant, but the 356/366 lines are very prominent and important for the UV curing process, since many photoinitiators have strong adsorption bands in this region.

High pressure Mercury Vapor Lamps: These lamps contain a mixture of mercury and xenon and operate above 10 atm. Under these conditions, the spectral output become a continuum one. These lamps

are normally small in dimensions, and used in photolithography.

### Metal Halide Lamps

In order to modify and adjust the spectral output and to meet with specific applications, other materials may be introduced into the tube. However, most of metals are less volatile than mercury and reactive to the electrodes or silica walls of the tube. Among others, metal halides (normally the iodide) can be used because of being volatile and not chemically active. For example, magnesium iodide lamps supply 280, 310 and 385 lines and improve cure speeds. Gallium iodide enhances the 400-450 nm region and is suggested to be suitable for curing pigmented finishes.

### Electrodeless Lamps

The electrodeless system activates the gas within the quartz bulb in a microwave field created by a small magnetron. The microwave energy (2450 Hz) excites the molecules within the tube very rapidly, and full power can be attained within 10 seconds, in contrast to the several minutes needed to start up electrode bulb. Most electrodeless bulbs are limited to 24 cm in length. For wider web processing, a series of these lamps are installed in a row.

Table 1 Comparison between Electrode and Electrodeless Lamps

	Conventional Electrode Lamp	Electrodeless Ultraviolet Lamp
Watt density:	120 watts/cm	120 watts/cm
Bulb length:	up to 2 meters	24 cm
Power source:	transformer and capacitor ballast	proprietary microwave system
Start-up time:	2 to 3 minutes	10 seconds
Stand-by condition:	half power	none required
Lamp warranty: (>85% output)	1,000 hours	3,000 hours

### Reflector System

Intense light may be desirable in high speed, high throughput operation. In such case, the lamp can be positioned at one focal point within an ellipsoid to concentrate ultraviolet light onto a target substrate. Since all UV sources also emit some visible and near infrared or heat energy, such light energy will also be concentrated at the focal point distant from an ellipsoidal reflector. In order to reduce this heat input, especially for heat sensitive substrates, a parabolic reflector in which the bulb is positioned at its focal point can be used. The parabola can be designed so that the light will be reflected outward in nearly parallel rays. Diffuse light is also desirable for dealing with shaped or three dimensional objects.

### Ancillary Equipment

#### Cooling

There is an emission of some infrared or heat energy from any type of light sources. The temperature of an UV source must be controlled not only because of the need to maintain controlled temperature conditions in and around the bulb, but also because of substrate considerations. Therefore, UV sources are usually kept cooled by the circulation of air or air and water.

#### Shuttering

It is essential to be able to cut off the source of UV at a very short notice e.g. in the event of a line stoppage. This is particularly important where flammable or heat sensitive materials are being used. In such situations it is necessary to fit a shutter to the UV assembly which will close automatically if the line is stopped.

#### Shielding

Exposure to UV radiation is extremely hazardous and must be avoided. It can cause temporary or even permanent blindness. Prolonged exposure to intense UV can result in skin cancer. Therefore, it is essential to have effective shielding with appropriate interlocks to prevent accidental exposure.

#3

# REGULATORY ASPECTS OF RADIATION USAGE



MY9700872

REGULATORY ASPECT OF RADIATION USAGE

ATOMIC ENERGY LICENSING BOARD

8 SEPTEMBER 1993



ATOMIC ENERGY LICENSING ACT, 1984  
( ACT 304 )

- . Being implemented on 1 February 1985
- . In general the purposes are:
  - to exercise control and supervision over the production, application and use of atomic energy
  - preparing Regulations
  - preparing Standard for liability for nuclear damage
  - other matters which are related to atomic energy
- . Explaining in general the matters must be obeyed by every "person" who exercise the activities related to atomic energy in Malaysia
- . Details explanation provided in the Regulations and Orders
- . Acts, Regulations and Orders must be obeyed by every "person" who are dealing with atomic energy and ionising radiations
- . Regulations and Orders are supported by other documents such as:
  - Code of Practices
  - Standards
  - Advice materials
- . The hierarchy for the Board's documents are as shown below:

ACT 304

REGULATIONS/ORDERS

CODE OF PRACTICES

ADVICE MATERIALS

## SUMMARY OF PROVISIONS UNDER ACT 304

Radiation Protection Act, 1986

## Section 12(1) (b)

- no person shall deal in, possess or dispose of any radioactive material, nuclear material, prescribed substances or irradiating apparatus, without having a valid licence issued under section 16 (5), Act 304.

- "deal in" means any activities related to radioactive material, nuclear material, prescribed substance or irradiating apparatus, involving the manufacturing, trading, producing, processing, purchasing, owning, using, transporting, transferring, handling, selling, storing, importing or exporting.

## Section 17

- conditions of licence
- provided with the issued licence
- can be added, varied or revoked by the appropriate authority

## Section 20

- every licensee who has in his possession or under his control any radioactive material, nuclear material, prescribed substance or irradiating apparatus shall make a return of such radioactive material, nuclear material, prescribed substance or irradiating apparatus to the appropriate authority as may be prescribed.
- the returns of possession shall be in the form prescribed in Radiation Protection ( Licensing ) Regulations, 1986. ( Form of returns of possession LPTA/BM/3)

## Section 21

- a return of every sale of any radioactive material, nuclear material, prescribed substance or irradiating apparatus shall be submitted to the Board.
- the returns of sale shall be in the form prescribed in Radiation Protection (Licensing) Regulation, 1986. (Form of return of sale LPTA/BM/2)

<b>POOR QUALITY ORIGINAL</b>
----------------------------------

. Section 22

- cancellation or suspension of licence
- The appropriate authority may at any time under any of the following circumstances in its discretion cancel or suspend for such period as it may think fit, any licence issued under this Act -
  - where the licensee has committed an offence under this Act
  - where the licensee has committed a breach of any of the conditions of licence
  - where the licensee ceases to work or operate the nuclear installation in respect of which the licence was issued; or
  - where in the opinion of the appropriate authority, it would be in the public interest so to do

. Section 24

- change of address
- every licensee shall obtain the prior approval of the appropriate authority in respect of any change of address of the premises specified in the licence.

. Section 25

- Radiation Protection ( details in Radiation Protection ( Basic Safety Standards ) Regulation 1988 )

. Section 33

- powers of search, seizure and arrest with warrant.

. Section 34

- powers of search, seizure and arrest without warrant
- the powers under this section include detain, inspect and arrest conveyance

. Section 35

- right of entry to any premises
- shall produce the letter of authorization or document of identification

. Section 39

- obstruction of inspection, search or investigation an offence
- liable to imprisonment for a term not exceeding 5 years; or
- fine not exceeding 5,000; or
- both

. Section 40

- any person who contravenes any of the provisions of this Act commits an offence under this Act-
- fine not exceeding 100,000; or
- liable to imprisonment for a term not exceeding 10 years; or
- both

**REGULATIONS**

- . Regulations are made by the Minister under section 68, Act 304.
- . Explaining the matters must be obeyed by every "person" who exercise the activities related to Atomic Energy.
- . 4 Regulations are :
  - Radiation Protection ( Licensing ) Regulations, 1986  
( P.U.(A) 149 )
  - Radiation Protection ( Basic Safety Standards )  
Regulations, 1988. ( P.U.(A) 61 )
  - Radiation Protection ( Transport ) Regulations, 1989  
( P.U. (A) 456 )
  - Atomic Energy Licensing ( Appeal ) Regulations, 1990  
( P.U.(A) 206 )

## RADIATION PROTECTION ( LICENSING ) REGULATIONS, 1986

- . Section 12 - No person shall deal in, possess or dispose of any radioactive material, nuclear material, prescribed substance or irradiating apparatus, without having a valid licence.
- . to get the licence, the Minister has produced the Radiation Protection ( Licensing ) Regulations, 1986.
- . This Regulation provide the methods and informations required for obtaining, renewal and amendment the licence from the Board.
- . Classification of licences
  - Class A ( radioactive material )
  - Class B ( nuclear material )
  - Class C ( irradiating apparatus )
  - Class D ( transportation )
  - Class E ( import/export )
  - Class F ( nuclear installation )
  - Class G ( dispose/store )
  - Class H ( for activities which are not covered by Classes A to G )
- . Licensing procedure in general
  - complete the application form
  - application fee ( 15.00/form )
  - Radiation protection officer
  - operators
  - details information of radiation workers
  - Radiation Protection Programme
  - instruments for personnel monitoring
  - radiation detection or measuring instrument ( survey meter)
  - details information of location for gauges

- details information of radiation instruments:
  - working manual
  - maintenance manual
  - engineering design for source container and transporting container
  - copies of certificate and standard
- the proposed design for storage
- approved registered medical practitioner ( ARMP )
- if the application is approved, fee licence must be paid before the licence is issued.

## RADIATION PROTECTION ( BASIC SAFETY STANDARDS ) REGULATIONS, 1988

- . Provide the regulations must be obeyed by every "person" who exercise the activities related to atomic energy.
- . the enforcement include transportation of radioactive material, nuclear material and prescribed substance.
- . System of Dose Limitation
  - justification
  - optimisation
  - annual dose limit
- . Exposure
  - special prohibition
    - i. no person shall allow any person under the age of eighteen years to work in a controlled area
    - ii. the person who has attained the age of sixteen years may be allowed to work in supervised area for training purposes only
    - iii. no person shall allow any person under the age of sixteen years to work in a supervised or a controlled area
    - iv. no person shall allow a female worker to work in a controlled area if she is or is suspected to be pregnant
    - v. no person shall employ a nursing mother in any work involving a high risk of radioactive contamination unless she is placed under special surveillance to detect any possible radioactive contamination.
  - Operational Radiation Protection
    - the licensee shall employ a radiation protection officer
    - classify the working areas into -
      - clean, supervised and controlled areas
      - ensure that supervised or controlled areas are clearly demarcated and appropriate and legible notices and warning signs bearing the radiation symbol are posted conspicuously in strategic places.



- the notice shall be in the national language, and in any other language if necessary
- ensure that operating instructions relevant to the supervised and controlled areas are posted conspicuously in such areas.
- monitoring of supervised and controlled areas
  - establish programmes for area monitoring in supervised and controlled areas.
- personnel monitoring in controlled areas
  - the doses received from external exposures shall be measured by the use of one or more approved personnel monitoring devices
  - the doses received from internal exposures shall be evaluated using techniques and procedures approved by the Board
  - inform each worker in writing of the worker's personnel monitoring results
- medical surveillance of workers
  - shall be carried out by an approved registered medical practitioner
  - shall include
    - pre-employment
    - periodic reviews of health
    - termination of employment or retirement
- maintenance of medical records of workers
  - medical record shall be kept up to date
  - confidential
  - shall retain the medical record of a worker in the form and manner acceptable to the appropriate authority

# RADIATION PROTECTION (TRANSPORT) REGULATIONS, 1989

- shall apply to transport of radioactive material, nuclear material or prescribed substance by all modes and land, water or in the air
- shall not apply
  - where radioactive material, nuclear material or prescribed substance are produced, used or stored other than storage in transit
  - to human beings who have been implanted with radioisotop cardiac pacemakers or other devices, or who have been treated with radiopharmaceuticals
  - to any radioactive material, nuclear material or prescribed substance which is an integral part of a conveyance
- classification of packagings
  - excepted packaging
  - type A packaging
  - type B packaging
  - industrial packaging
- the use of non-conforming packaging
  - conformity with the relevant requirements of these Regulations is in the opinion of the Board impacticable
  - suitable measures to compensate for the known or the anticipated non-conformities are available and the Board believes that the overall levels of safety in transport and in stowage is at least equivalent to that which would have been provided if all the relevant requirements of these Regulations had been met.
- prohibition
  - no person shall transport or cause to be transported any radioactive material, nuclear material or prescribed substance unless -
    - it is packed in a packaging whose design meets all the requirements of these Regulations
    - the consignment is packed marked, labelled, categorised and placarded in accordance with all the requirements of the Regulation.

# ATOMIC ENERGY LICENSING ( APPEAL ) REGULATIONS, 1990

- Being implemented on 26 July 1990
- provide the matters must be obeyed by every "person" who want to make an appeal against any decision of the appropriate authority
- every appellant shall -
  - state precisely in the notice of appeal the decision with which he is dissatisfied
  - state in the notice of appeal his full name and address
  - send the notice of appeal so as to reach the Minister within the time stated in subsection (1) of section 32 of the Act; and
  - send a copy of the notice of appeal to the appropriate authority so as to reach the appropriate authority within the time stated in subsection (1) of section 32 of the Act for the giving of such notice to the Minister.
  - every notice or other document relating to an appeal shall be sent by registered post.

## ORDERS

- ATOMIC ENERGY LICENSING ( EXEMPTION ) ( SMOKE DETECTORS ) ORDER, 1989
- ATOMIC ENERGY LICENSING ( EXEMPTION ) ( LIGHTNING ARRESTER ) ORDER, 1990
- ATOMIC ENERGY LICENSING ( EXEMPTION ) ( LEASING AND HIRE - PURCHASE ) ORDER, 1990
- EXCLUSIVE ECONOMIC ZONE ( APPLICATION OF ATOMIC ENERGY LICENSING ACT, 1984 ) ORDER 1990

**#4**

**UV/EB CURING OF SURFACE  
COATINGS: WOOD-BASED  
SUBSTRATES**



## UV/EB Curing of Coating of Wood Substrate

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CHUGOKU MARINE PAINTS, LTD.  
YASU-CHO, SHIGA, 520-23. JAPAN

Radiation curing has found many applications in recent years, due to the advantages, mainly high curing speed and good film performances. The use of UV curing started commercially 20 years ago, but it's in the last 10 years that the application for wood coating was increased in Japan. Big market of UV wood coating is plywood for flooring. Many manufacturers of plywood in Japan changed conventional coating lines into UV coating lines in this decade. In the future, 3-dimensional UV coatings for wood substrate, such as joineries, will be common by introducing of vacuum coating. On the contrary, EB curing for wood substrates is uncommon in Japan.

### 1. UV Coating

The UV curable coating is mainly made of resin, monomer and photoinitiator.

Resin	: 100 % solid low molecular weight oligomer.
Monomer	: High boiling point reactive diluent which reduce viscosity.
Photoinitiator	: This chemical absorbs the radiation energy and produces reactive species that initiate polymerization of resin and monomers.
	Photoinitiator is not required in EB curing.

Formulations commonly contain other components such as :  
fillers, additives, flatting agents.

Fig 1, 2<sup>1)</sup> shows the composition of UV coating and the reaction of UV/EB curing schematically.

### 2. Comparison of Curing Method

One of the advantages of radiation curing is solvent-free. Conventional coatings are produced by mixing components with a solvent, and this solvent must be evaporated to cure the coating by thermal-heating devices. On the other hand, UV/EB formulations are 100 % solid system, with avoidance of solvent emission and reduction of air pollution. Although UV/EB coating may cost more than twice compared with conventional coating, costs on a solid basis are usually comparable. Table 1. shows the comparison of curing methods.

### 3. Wood Coating

UV coatings are commonly accepted as an efficient and economical method for wood industry in Japan.

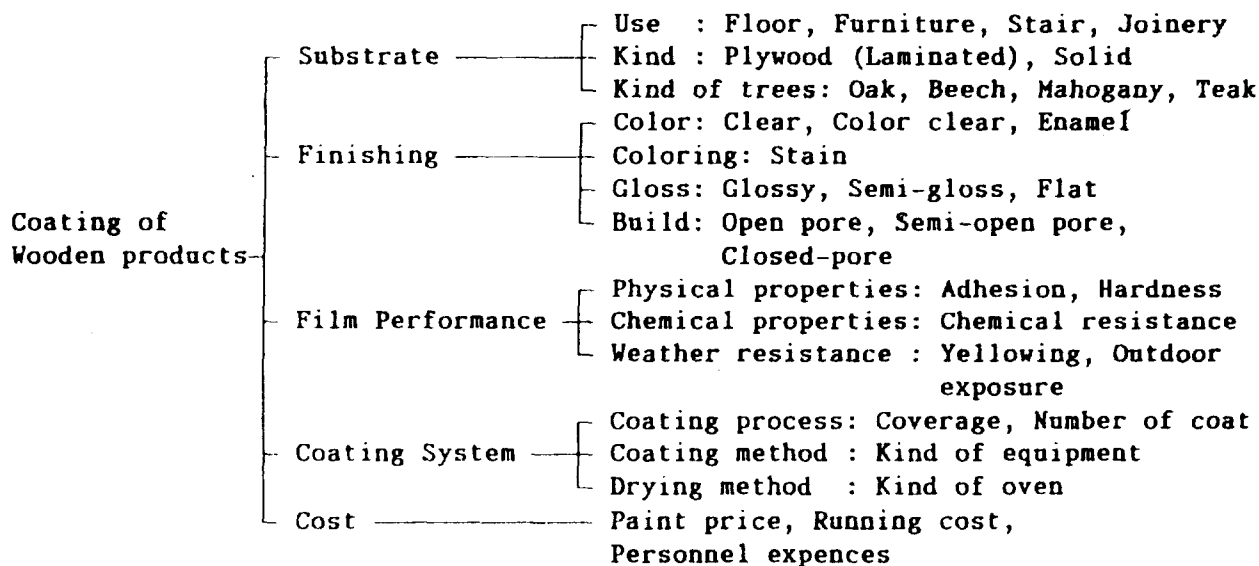
UV systems used for wood coating are based on unsaturated polyesters or acrylic resins. Although acrylic UV coatings provide excellent properties and faster curing speed, they are more expensive than unsaturated polyesters.

Characteristic of unsaturated polyesters and acrylics is shown in Table 2.

UV coatings are applied on wood substrates by such coating method :

- ◆ Roll Coat (Natural, Reverse)
- ◆ Curtain Coat
- ◆ Spray
- ◆ Vacuum Coat

Coating design for wooden products is shown below.



Sales volume of UV coatings in Japan is shown in Table 3<sup>2)</sup>.

### 4. UV Coating System for Color Flooring in Japan

One of the biggest market of wood coating is flooring.

During the last 10 years, great progress has been achieved in this field by UV curable systems; due to high curing speed, 100 % solid and good film performances.

Finally, a typical UV coating system of color flooring in Japan is shown in Table 4<sup>3)</sup>.

### REFERENCES

- 1) SASAKI, T., TE2. Radiation Processing of Polymers.
- 2) YAMAGUCHI, I., Data from Fuji Merchandising Center.
- 3) YAMAKAWA, M., Toso Gijutsu, November 1991, 83-86.

FIG.1 Composition of UV Curable Coatings

Oligomer

Monomer

Photoinitiator

(Not required in EB)

Fillers, Additives, Flatting Agents

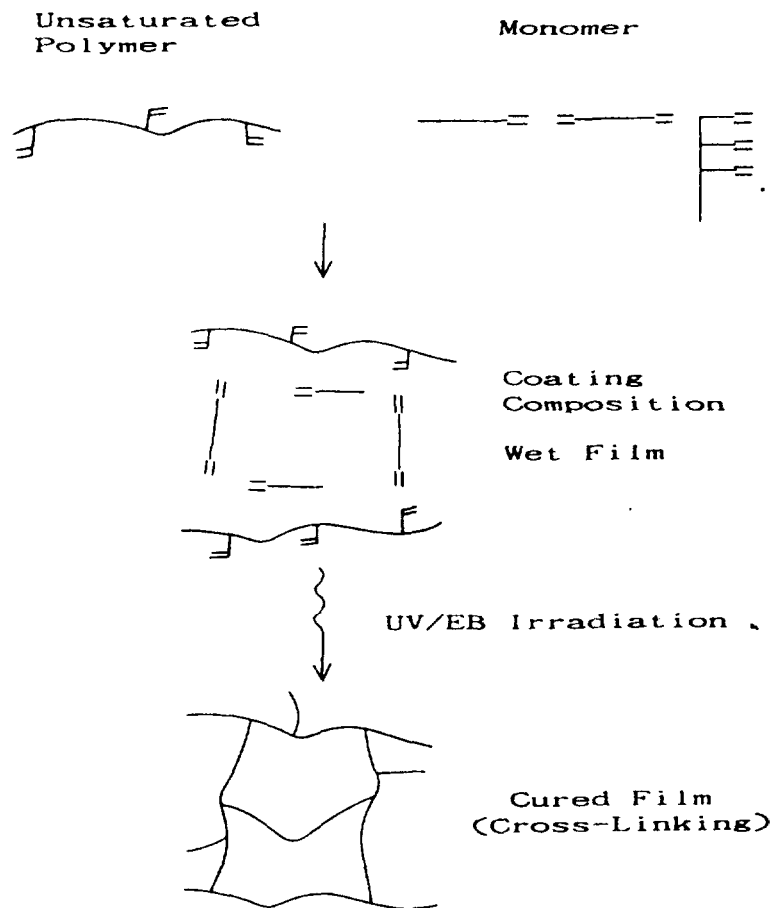


FIG.2 Schematic Diagram of UV/EB Curing



Table 1. Comparison of Curing Methods

Items	UV Curing	EB Curing	Baking (Conventional)
Initial cost for installation	Low	High	Middle
Length of oven (meter)	5	3	30 - 100
On-Off operation	Possible	Possible	Impossible
Special atmosphere	Not necessary	Necessary	Not necessary
Curing time	Several seconds	Less than 1 seconds	Several minutes - several ten minutes
Compatibility to non-heat-resistant substrates	OK	OK	No
Pigmentation	Limited	OK	OK
Catalizer	Necessary	Not necessary	Both necessary & not necessary
Solid content (%)	100	100	35 - 65
Thinning ratio (%)	0 - 30	0	35 - 65
Precautions for worker's health	UV rays Ozone	X rays Ozone	Heat Evaporated solvents
Cost for enviromental problems	Low	Low	High

Table 2. Characteristics of Oligomer

Oligomer	Structure of Oligomer	Characteristic
Unsaturated Polyester	$\text{-(C=C-C-O-C=C)-}$ $\quad \quad \quad \parallel$ $\quad \quad \quad \text{O}$	Low-price, Slow curing speed
Urethane Acrylate	$\text{C=C-(N-C)-C=C}$ $\quad \quad \quad   \quad \parallel$ $\quad \quad \quad \text{H O}$	High-price, Flexibility
Epoxy Acrylate	$\text{C=C-(C-C-O-Ph-C-Ph)-C=C}$ $\quad \quad \quad  $ $\quad \quad \quad \text{OH}$	Chemical resistance Rapid cure
Polyester Acrylate	$\text{C=C-(C-O)-C=C}$ $\quad \quad \quad \parallel$ $\quad \quad \quad \text{O}$	Stain resistance Low viscosity

Table 3. Sales Volume of UV Coating in Japan

	(tons)			
	1988	1989	1990	1991
Hard Coating	600	720	860	1,030
Wood Coating	1,600	2,100	2,700	3,500
PVC Flooring	700	750	830	950
Metal Coating	255	270	280	290
Plastic Film	90	100	110	120
Total	3,245	3,940	4,780	5,890

Table 4. UV Coating System of Color Flooring

Process	Paint(CMP)	Coating Equipment	Coverage (g/ft <sup>2</sup> )	Curing (30m/min)
1. Staining	STAIN N	S-R-N Roll Coater	1~2	80°C×90sec
2. Sealer	AULEX NO.800S	N Roll Coater	1	UV 3 lamps
3. Undercoat	AULEX NO.630	N-N Roll Coater	2	UV 6 lamps
	WA-500			
	GC-500			
4. Sanding	-	Wide Belt Sander	-	-
	① AULEX NO.630	N-N Roll Coater	1.5	UV 8 lamps
5. Topcoat	② AULEX NO.655	Curtain Coater	6	80°C×90sec UV 6 lamps
	③ MARBLAC OL	Curtain Coater	6	60°C×15min

WA-500,GC-500: Abrasive. S: Sponge. N: Natural. R: Reverse

MARBLAC: Acid curing amino-alkyd resin paint.

**#5**

**UV/EB CURING OF SURFACE  
COATINGS: NONWOOD-BASED  
SUBSTRATES**

**WORKSHOP ON SURFACE FINISHING BY RADIATION**  
**CURING TECHNOLOGY**

**UV – EB Curing of Surface Coatings**

**8 – 9 September 1993**  
**Unit Tenaga Nuklear, Bangi**

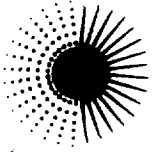
**Jean-Pierre Ravijst**  
Business Development Manager  
South East Asia



**Chemicals**

**UCB s.a. - Representative Office**  
Orchard Road 390  
Palais Renaissance # 09-03  
0923 Singapore  
Tel. (65)7350949  
Fax (65)7350946

# MARKET EVOLUTION



**Radcure**

**SIXTIES:**

Unsaturated polyester in styrene

--->

wood coatings

**SEVENTIES:**

Acrylates

--->

Graphic Arts

paper/plastics

--->

Paints & Coatings

wood

plastics

--->

Electronics

solder resist

etch resist

**EIGHTIES:**

Variety of applications

--->

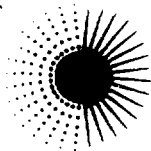
Increase use of ELECTRON BEAM

--->

3 D substrates

--->

Non radiation cure applications



**Radcure**

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## MARKET OVERVIEW BY APPLICATION

### 1. GRAPHICS ARTS / paper-plastics-metal

advantages:

solventless  
high cure speed  
chemical resistance  
smoothness

### 2. COATINGS clear/pigmented -- UV or EB cure

advantages:

chemical resistance  
reduced energy cost  
no solvent emission  
increased productivity  
low capital investment (for UV-equipment)  
low equipment space required  
long potlife of 1K-systems  
thin coating thickness

general disadvantages:

oxygen inhibition (EB-cure)  
adhesion on certain substrates  
    due to the shrinkage  
potential for irritancy and  
    sensitization

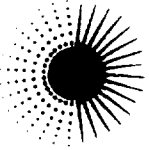
## 24 HOURS A DAY WE ARE CONVOQUED

## WITH OBJECTS WHERE RADIATION CURE

## AT HOME

- cosmetic packaging
- tubes (toothpastes) . primary packaging  
  . secondary packaging
- drug packaging (blisters, boxes)
- dental applications

- 3 D furniture
- kitchen furnitures/cabinets
- ceramics plates
- magazines
- book covers
- flooring (wood/PVC)
- wooden frames/doors
- walls/panels
- safety glass
- electrical installation
  - \* radio
  - \* tv
  - \* telephone
  - \* CD's
- building panels

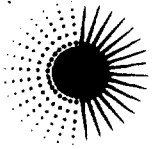
**Radcure****PLEASURE**

- cars (frames, headlamps, electrical connenctions, windshields)
- ski
- golf balls
- helmets
- fishrods
- boats

**FURTHER**

- labels (adhesives)
- bottles (plastic, glass)
- bank notes, credit cards
- cans (beer, cola...)
- packaging: papers  
                  plastics  
                  aseptic



**Radcure**

## EPOXY ACRYLATES

used in Radiation cure application

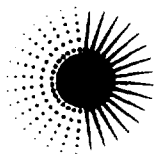
### 1] BY CURE SYSTEM

UV - COATINGS	62 %
EB - COATINGS	5 %
UV - INKS	9 %
EB - INKS	1 %
ADHESIVES	3 %

---

### 2] BY APPLICATION

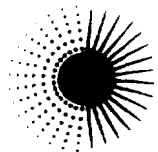
WOOD	6 %
PAPER	69 %
PLASTIC	10 %
METAL	13 %
OTHERS	2 %

**Radcure**

## URETHANE ACRYLATES

used in Radiation cure application

WOOD	11 %
PAPER	6.5 %
PLASTIC	74.5 %
METAL	1.5 %
<hr/>	
OTHERS	6 %



## **NEW APPLICATIONS IN DEVELOPMENT**

### **APPLICATIONS AND PRODUCTS IN DEVELOPMENT**

- LAMINATING ADHESIVES
- PRESSURE SENSITIVE ADHESIVES
- ABRASIVE PAPER
- TEXTILE COATINGS
- OUTDOOR RESISTANT COATINGS
- COATINGS FOR FOOD CONTACT
- PIGMENTED UV-CURABLE COATINGS
- LOW VISCOUS OLIGOMERS
- CLEANER PRODUCTS (LESS IRRITANT OR LESS TOXIC)

# GUIDELINES FOR SUGGESTED USES

Business Unit UCB Radcure



**POOR QUALITY  
ORIGINAL**

EB CURING	SPECIAL CHARACTERISTICS	PREPOLYMERS & ADDITIVES	MONOMERS & DILUTING OLIGOMERS
-----------	-------------------------	-------------------------	-------------------------------

**LITHOGRAPHIC  
INKS**

High reactivity, good HLB,  
low extractables, low odour,  
low misting

Eb 1657, Eb 1870

Eb 53  
Eb 1140

**OVERPRINTING  
VARNISHES**

High reactivity,  
low odour, low extractables

Eb 80, Eb 1608, Eb 1810  
<Eb 1360>

TPGDA (DEO)  
Eb 53

**WOOD  
AND BOARD**

Abrasion, stain resistance  
Through cure of pigmented  
coatings

Eb 80, Eb 82, Eb 284, Eb 505  
Eb 810, Eb 830  
Eb 605, Eb 1608

TPGDA  
OTA 480

**PAPER  
UPGRADING**

Abrasion and scratch  
resistance  
Metalization

Eb 605, Eb 810  
<Eb 1215>  
Eb 1755

TPGDA  
OTA 480

**OUTDOOR  
RESISTANT  
COATING**

Good weatherability

Eb 284

HDDA  
TPGDA

**LAMINATING  
ADHESIVES**

Adhesion, cohesion,  
flexibility  
Through cure of opaque  
substrates

Eb 230, Eb 525, Eb 604  
Eb 810, Eb 830, Eb 1810

HDDA  
TMPTA  
TPGDA (DEO)

UV CURING	SPECIAL CHARACTERISTICS	PREPOLYMERS & <ADDITIVES>	MONOMERS & DILUTING OLIGOMERS	PHOTOINITIATORS
LITHOGRAPHIC INKS	High reactivity Good HLB, low misting  Improved reactivity Improved pigment wetting	Eb 436, Eb 438, Eb 3436, Eb 3438 Eb 657, Eb 860, Eb 870 Eb 1608, Eb 1870 Eb 3200, Eb 3608 <Eb 220> <Eb 3201>	Eb 40 Eb 140 Eb 150 OTA 480	BENZOPHENONE IRGACURE 369 IRGACURE 651 IRGACURE 907 QUANTACURE ITX QUANTACURE DMB UV 20
FLEXO INKS	High reactivity, low viscosity	Eb 80, Eb 81, Eb 82 Eb 810	DPGDA, HDDA TPGDA OTA 480	BENZOPHENONE Eb P 36, Eb P 115 Eb 7100
SILK SCREEN INKS	Good rheology	Eb 210, Eb 436, ... Eb 605, Eb 745, ... Eb 3702, Eb 4858, Eb 8800	HDDA TPGDA Eb 2047	IRGACURE 651
OVERPRINTING VARNISHES	High reactivity	Eb 210, Eb 264 Eb 436, Eb 584 Eb 605, Eb 1608, Eb 3600 Eb 80, Eb 81, Eb 82, Eb 745, Eb 810 <Eb 350>	HDDA, TMPTA TPGDA OTA 480	BENZOPHENONE DAROCUR 1173 Eb P 36 Eb P 115 IRGACURE 184 IRGACURE 651
WOODFINISHING				
Sealers and fillers	Sandability, wetting	Eb 80, Eb 505, Eb 605, Eb 608	TPGDA, OTA 480	BENZOPHENONE DAROCUR 1173 Eb P 115 Eb 7100 IRGACURE 184 IRGACURE 651
Varnish	Hardness	Eb 80, Eb 81, Eb 82, Eb 810, Eb 1608, Eb 3500	HDDA, TMPTA TPGDA, OTA 480	
Parquet	High abrasion resistance	Eb 264, Eb 294, Eb 810	HDDA	
Outdoor	Non-yellowing and outdoor durability	Eb 284, Eb 285, Eb 8804	HDDA	BENZOPHENONE DAROCUR 1173 IRGACURE 184 QUANTACURE ITX
Pigmented coatings	Reactivity, in-depth cure	Eb 604, Eb 810, Eb 1290	HDDA	
ELECTRONICS				
Solder resist	Soldering resistance	Eb 616, Eb 629, Eb 639, Eb 1259, Eb 3604 <Eb 169>	Eb 109 TMPTA	BENZOPHENONE DAROCUR 1173 ETHYLANTRAQUINONE*
Etch resist	Acid resistance Easily stripped in alkaline solution	Eb 770, Eb 1559 <B-CEA>	Eb 109 HEMA	IRGACURE 651
* only for solder resist				
COATINGS ON PVC, LINOLEUM	Abrasion resistance	Eb 204, Eb 205, Eb 245, Eb 264, Eb 265, Eb 284, Eb 285, Eb 294, Eb 295	HDDA TPGDA	BENZOPHENONE DAROCUR 1173 Eb P 115, Eb 7100 IRGACURE 184 IRGACURE 651
COATINGS ON OTHER PLASTICS	Adhesion	Eb 245, Eb 436, Eb 525, Eb 584, Eb 585, Eb 745, Eb 769, Eb 1701 Eb 3604, Eb 3701 Eb 4244, Eb 5129, Eb 8800 <Eb 350>	HDDA IBOA PETIA TPGDA	BENZOPHENONE DAROCUR 1173 IRGACURE 184 IRGACURE 651
CORK	Abrasion resistance with moderate flexibility	Eb 210, Eb 204, Eb 264, Eb 284	HDDA - ODA OTA 480	BENZOPHENONE DAROCUR 1173 IRGACURE 184 IRGACURE 651
METALLIZED SUBSTRATES	Adhesion, protection	Eb 745, Eb 754, Eb 1629 <Eb 169>, <Eb 170> <B-CEA>	HDDA IBOA TPGDA	BENZOPHENONE DAROCUR 1173 Eb P 36 IRGACURE 651
METALS	Adhesion, protection	Eb 210, Eb 284 Eb 604, Eb 629 Eb 524 <Eb 169>, <Eb 170>, <B-CEA>	HDDA IBOA TMPTA TPGDA	BENZOPHENONE DAROCUR 1173 Eb P 36 IRGACURE 651
LAMINATING ADHESIVES	Adhesion, cohesion, flexibility	Eb 230	HDDA	BENZOPHENONE

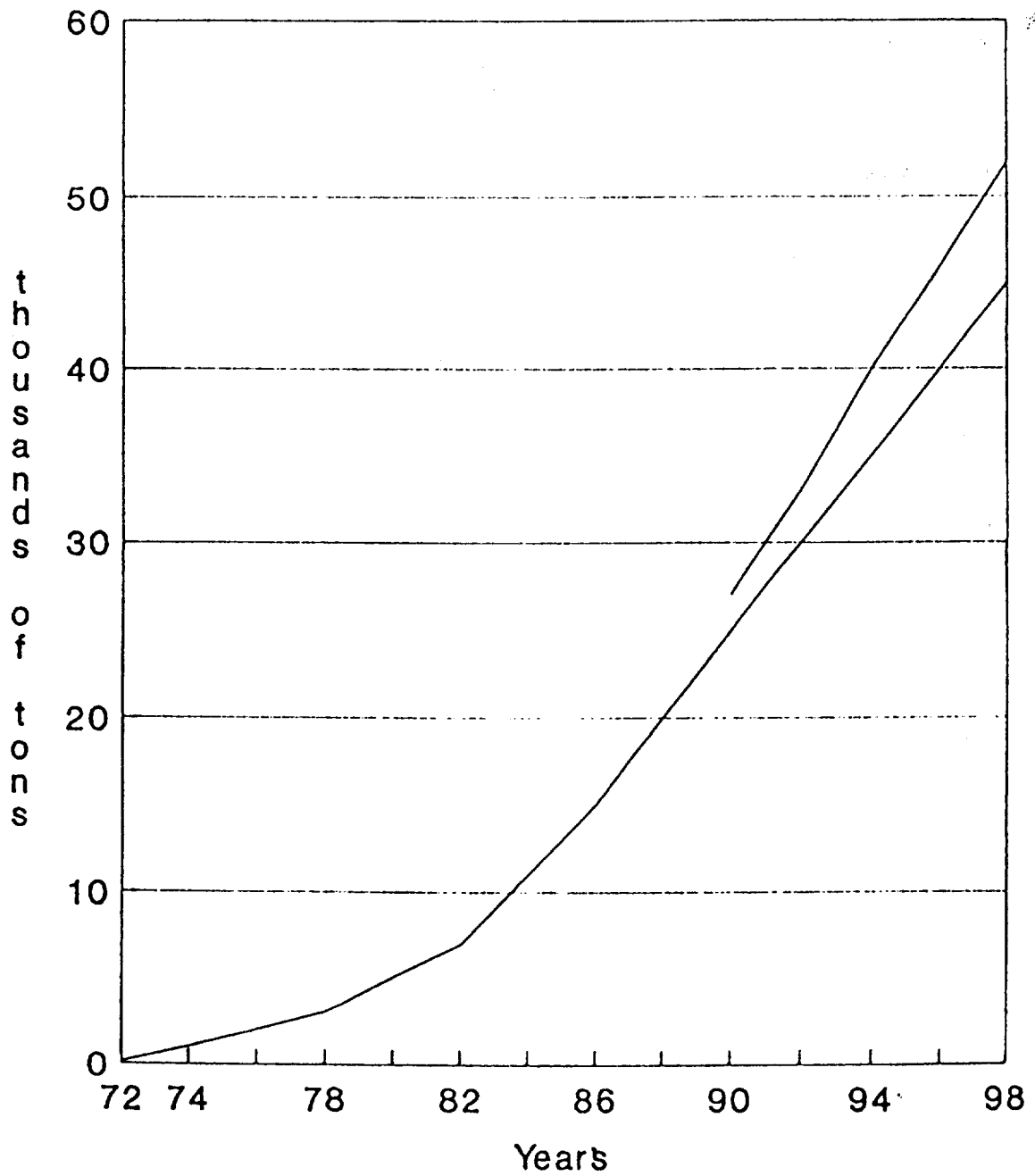
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**Radcure**

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## Evolution of European Market Acrylated Products



# EUROPEAN MARKET OVERVIEW

## BY APPLICATION

### 1. GRAPHICS ARTS

/ paper-plastics-metal

OFFSET INKS AND VARNISHES

SCREEN PRINT INKS AND VARNISHES

FLEXO INKS

### 2. COATINGS

clear/pigmented -- UV or EB cure

#### A. Paper:

paper upgrading

metallization

decor paper

wood imitation

release coatings

building panels

#### B. Plastics:

-PSA

- laminating adhesives

-floor coverings (PVC-flooring)

-automotive (headlights, covers --> PC

-compact disks (PC)

-skies

-wood imitated PVC for furniture

-leather & artificial leather

#### C. Wood:

-pigmented coatings on fiber board

-parquet flooring

-kitchen cabinets

- 3D parts (spray applications)

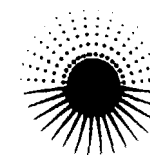
-hardboard (doors)

#### D. Metal:

-galvanized tubes

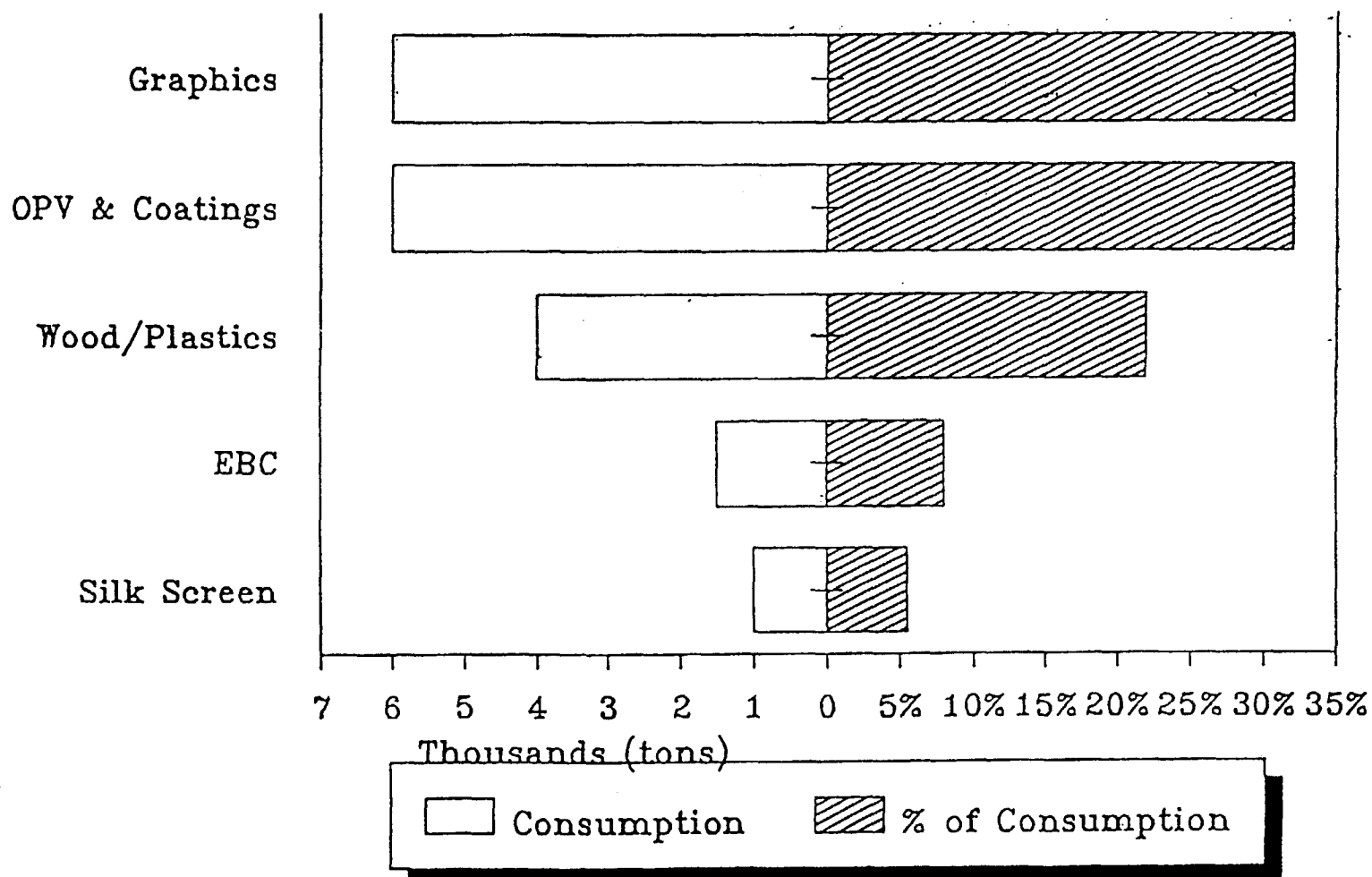
-metallic furniture

# CONSUMPTION BY APPLICATION FIELD



**Radcure**

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ESTIMATION OF VOLUME (TONS) OF RADIATION CURE PRODUCTS USED IN THE INDUSTRY
--

	<u>European Market</u>	<u>US Market</u>
<u>GRAPHIC ARTS</u>	14600	11200
- LITHO	5000	2800
- SCREEN	1100	1400
- OPV	8500	7000
<u>COATINGS</u>	7100	8300
- WOOD	5000	3500
- PLASTIC	1000	2500
- METAL	400	1100
- PAPER	700	1200
<u>ADHESIVES</u>	400	800
<u>ELECTRONICS</u>	900	700
CATIONIC	200-500	300
POLYESTERS	15000	12000

**Radcure**

# ACRYLATED BINDERS

	<u>European Market</u>	<u>US Market</u>
MONOMERS	11500	9600
PHOTOINITIATORS	1500	1300
EPOXY ACRYLATES	4500	4700
URETHANE ACRYLATES	2500	3100
POLYESTER ACRYLATES	3500	800
FULL ACRYLATES		800
	<hr/>	<hr/>
	23500	20300

**Radcure**

## MONOMERS / LOW VISCOSITY OLIGOMERS

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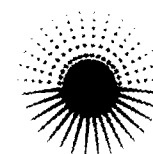
- POLY FUNCTIONAL		300 T
- TRIFUNCTIONAL		3900 T
- DIFUNCTIONAL		3900 T
- MONOFUNCTIONAL	- ACRYLATES	800 T
	- METACRYLATES	800 T

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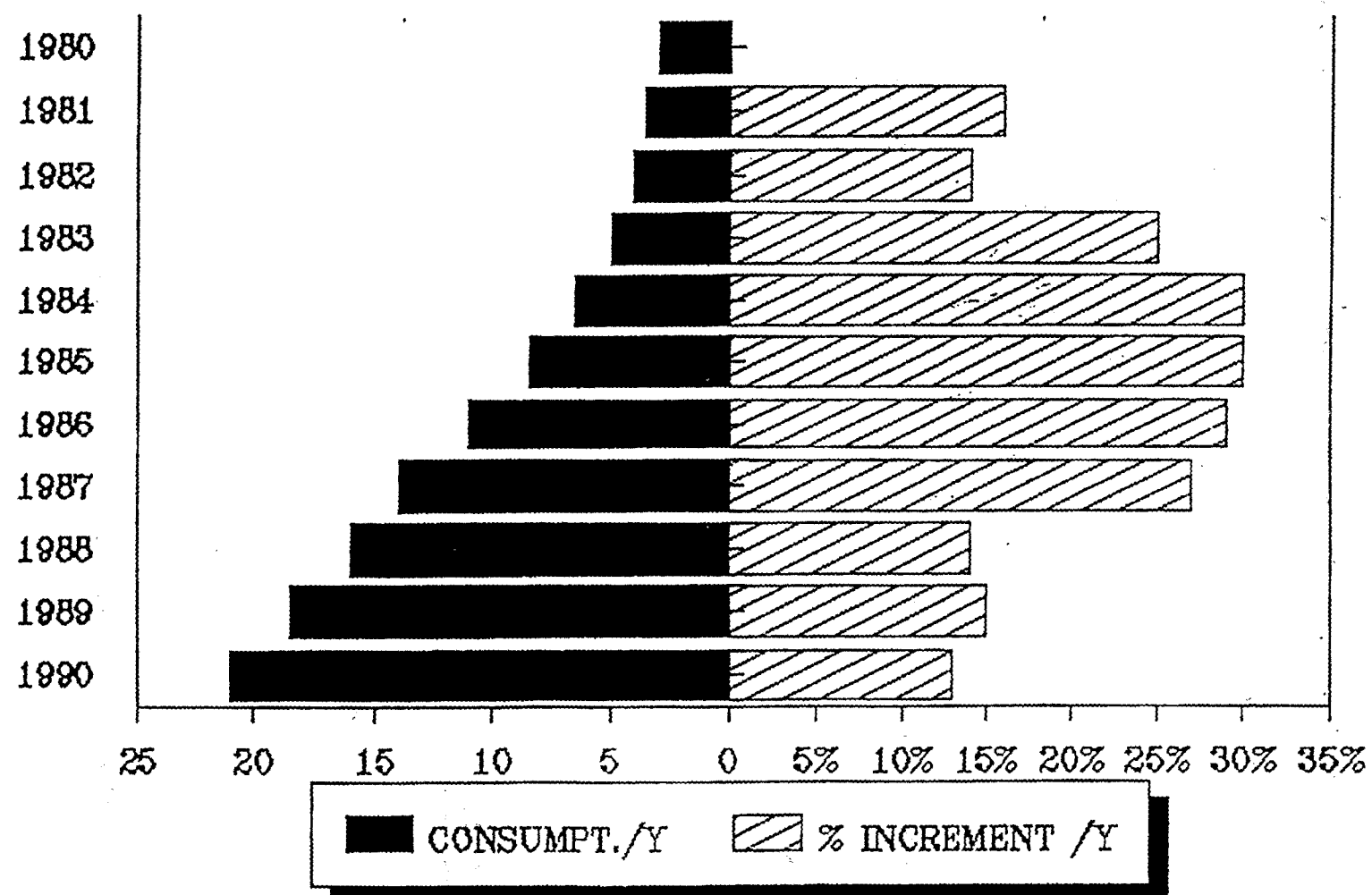
## OTHERS

PHOTOINITIATORS :	- FREE /RADICAL	1000 T
	- CATIONIC	15 T
PLASTICIZERS + PIGMENTS + FILLER + ETC..		2300 T

# MARKET GROWTH ESTIMATION



**Radcure**



#6

# DEVELOPMENT OF EPOLA AND ITS APPLICATION



# THE DEVELOPMENT OF EPOXIDISED PALM OIL ACRYLATE (EPOLA) AND ITS APPLICATIONS

by  
**Mohd. Hilmi Mahmood**  
**Radiation Processing Program**  
**Unit Tenaga Nuklear, UTN**

## INTRODUCTION

In recent years, there are growing trends in using vegetable oils as raw materials in resin production. The reasons towards moving into this direction are, stricter environmental legislation towards maximum allowable volatile organic contents (VOC), preserving the earth's natural resources and less accumulation problems in the environment.

Vegetable oils (e.g. soybean, linseed, tung, sunflower, corn, palm, cotton etc.) are chemically known as glycerol triesters of polyunsaturated fatty acids or unsaturated triglyceride oils. They are products of domestic agriculture and are, hence, renewable. Their supply is plentiful and therefore, they are inexpensive. These oils are quite different from each other. They are categorised into three groups : drying oils, semi-drying oils and non-drying oils ; or by their power to absorb oxygen from surrounding air, which is directly proportional to the iodine value of the oil which measure the extent of unsaturation of the fatty acids present. The fatty acids are characterised as saturated or unsaturated. The more unsaturated the oil, the faster it will react with oxygen and polymerise.

**Table 1 : Typical structures of Unsaturated Triglyceride Oils**

Name	Structure
Soybean oil	$\text{CH}_3-(\text{CH}_2)_4-\text{CH}=\text{CH}-\text{CH}_2-\text{CH}=\text{CH}-(\text{CH}_2)_7-\text{CO}_2-\text{CH}_2$
	$\text{CH}_3-(\text{CH}_2)_4-\text{CH}=\text{CH}-\text{CH}_2-\text{CH}=\text{CH}-\text{CH}_2-\text{CH}=\text{CH}-(\text{CH}_2)_4-\text{CO}_2-\text{CH}$
	$\text{CH}_3-(\text{CH}_2)_7-\text{CH}=\text{CH}-(\text{CH}_2)_7-\text{CO}_2-\text{CH}_2$
Palm oil	$\text{CH}_3-(\text{CH}_2)_7-\text{CH}=\text{CH}-(\text{CH}_2)_7-\text{CO}_2-\text{CH}_2$
	$\text{CH}_3-(\text{CH}_2)_4-\text{CH}=\text{CH}-\text{CH}_2-\text{CH}=\text{CH}-(\text{CH}_2)_7-\text{CO}_2-\text{CH}$
	$\text{CH}_3-(\text{CH}_2)_{13}-\text{CO}_2-\text{CH}_2$

Tung and linseed (nine unsaturation) oils have been formulated into lithographic varnishes for a long time because they dried fast and hard. Linseed and soybean (six unsaturation) oils are widely used in alkyd resins, which are used in dispersion and flushing vehicles. Soybean oil which is well known for its colour retention and low odour properties, has also been widely used in napkin and metal decorating inks (Chris Halvorsen, 1992).

**Table 2 : Fatty Acids composition (%) of vegetable oils.**

Name	linseed	soybean	palm	number of carbons	number of double bonds
Oleic	17	24	43	18	1
Linoleic	14	51	11	18	2
Linolenic	60	9	0.4	18	3
Palmitic	6	12	40	16	0
Stearic	5	4	4.4	18	0

**Table 3 : Prices of selected oils and fats, 1987-1990 (U.S.\$/ton).**

Product	1987	1988	5/89	7/90
Soybean oil, Dutch, fob ex-mill	334	463	460	434
Coconut oil, Phil/Indo. cif Rotterdam	442	565	589	330
Palm kernel oil, Malaysia, cif Rotterdam	426	539	528	335
Tallow, U.S bleach-fancy, cif Rotterdam	356	412	369	345

Source : Oil World.

World **palm oil** (*Elaeis guineensis*) production grew from 1.7 million metric ton in 1970 to 10 million metric ton in 1990 for an average annual increase of 9.3%. Malaysia provides 57% of the world's production. Malaysia's role as an exporter is even greater, accounting for 75% of world exports. Indonesia, Papua New Guinea, and the Ivory Coast account for an additional 20% of the world production and export (Kaufman *et. al.*, 1990).

**Table 4 : Average annual yields of selected vegetable oils.**

Oil	Annual yield (Kg/hectare)
Palm oil	4000-5000
Palm kernel oil	400-500
Coconut oil	710
Soybean oil	389
Peanut oil	875

**Table 5 : World production of selected oils and fats as raw materials for oleochemicals (million ton)**

Oils & fats	1950	1960	1970	1980	1989	1990f	1995f	2000f
Soybean	2.1	4.0	6.1	12.2	15.0	16.1	18.2	21.0
Tallow	2.2	3.6	4.4	6.0	6.6	6.6	7.1	7.0
Coconut	1.9	2.1	2.2	3.3	2.8	3.1	3.8	4.2
Palm	0.9	1.1	1.7	5.0	10.3	10.8	17.8	22.0
Palm kernel	0.4	0.4	0.4	0.7	1.3	1.4	2.3	2.8
Others	16.1	20.9	25.3	29.6	41.3	41.2	35.2	50.0
Total	23.6	32.1	40.1	56.8	77.3	79.2	84.4	107.0

f: forecast.

Source : 1950 to 1995f - Oil World.  
2000f - Estimated.

#### PRODUCTION OF RADIATION CURABLE RESIN AND ITS APPLICATIONS

Palm oil and its products (palm olein and stearin) contain level of unsaturation that are half or less than half of that of soybean oil. It has therefore never been thought of as suitable raw materials for the production of resins. However it was of interest to find out to what extent could palm oil or its products compare with linseed oil, soybean oil etc. As even a slight substitution is a step towards changing the situation from having to import to being self-sufficient.

For the last three years, one of the main research activities in radiation processing laboratory, UTN, has been in modifying materials from indigenous natural sources particularly palm oil products into radiation curable oligomers/resins, namely **acrylated oils**. Epoxidation of palm oil products has been reported to yield materials namely epoxidised palm oil products (EPOP) suitable as plasticiser and stabiliser for plastics (Salmiah *et. al.*, 1987). Acrylated palm oil is prepared through **acrylation process**, whereby, acrylic acid is introduced into **oxirane** group of the EPOP, the process is similar to those used to epoxy acrylate manufacture (Hussin *et. al.*, 1990). The acrylated products called epoxidised palm oil products acrylate, **EPOLA**, was found curable when subjected to ultraviolet (UV) light giving soft coatings. Addition of crosslinkers such as difunctional and trifunctional acrylates enhanced the physical properties of coatings. Polyurethane acrylate (PUA) can also be added into formulation to improve properties particularly flexibility and toughness (Mohd. Hilmi *et. al.*, 1991). Acrylated oils are having advantages of excellent pigment wetting, low cost, good adhesion and low skin irritancy, but of the disadvantages of slow cure and soft film (for surface coatings). Later it was discovered that EPOLA based formulations (with the incorporation of other oligomers and monomers) could satisfactorily be coated on wood substrates (rubberwood parquets) without any major defects. Their properties were almost comparable with commercial resins and possess a great potential to be used for radiation curable finishes (Mohd. Hilmi *et. al.*, 1992).

The use of acrylated epoxidised palm oil products (EPOLA) for other applications such as radiation curable filler/sealer and radiation curable pressure sensitive adhesives has also been ventured. Early results showed that EPOLA is also promising to be used in both applications.

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#7

# DEMONSTRATION OF UV/EB PROCESS



**National Workshop on Surface Finishing  
by  
Radiation Curing Technology**

*8th - 9th September 1993, Bangi, Selangor D.E.*

**DEMONSTRATION and PRACTICAL EXERCISES on  
RADIATION CURING TECHNOLOGY**

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Japan International Cooperation Agency (JICA) and  
International Atomic Energy Agency (IAEA).**

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## **Part I            DEMONSTRATION**

### **Application for Ultraviolet (UV) and Electron Beam (EB) Curing**

#### **a.        Substrate**

Plywood will be used in this demonstration.

#### **b.        Coating Materials**

##### **i.        UV Curing of Basecoat :        Reverse Roll Coat Filler**

This formulation can be applied directly to wood i.e. to fill the pores of the substrate in order to obtain a smooth surface for subsequent coating operations.

##### **ii.       EB Curing of Topcoat :        Curtain Coating Acrylic Topcoat**

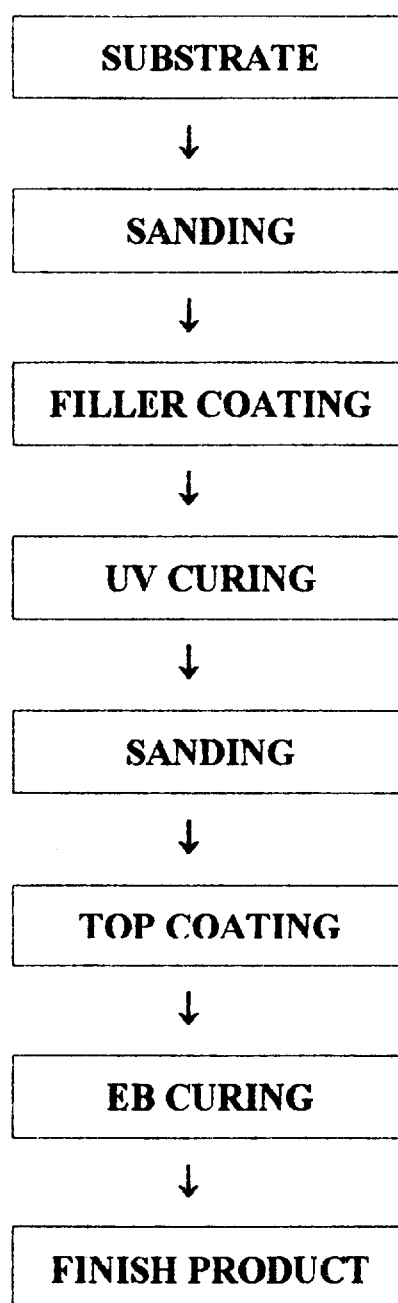
The final topcoat will be applied by using curtain coater (in some instances there are two final topcoat applications with the second coat being a clear coat, with potential for as many as five final coats when seeking extremely high-gloss finishes). A 200 keV linear filament accelerator from NHV is used. This voltage is sufficient to penetrate deep into the radiation curable coating materials ensuring complete cure of filler/sealer and topcoats.

#### **c.        Experimental Procedure**

1. Take a piece of wood panel and sand it to give a smooth surface.
2. Coat the sample with filler/sealer using automatic reverse roller coater (Appendix I).
3. Irradiate the above sample using ultraviolet irradiator (Appendix III) and sand it again.

4. Cut the sample into six or eight pieces of size 10 cm x 15 cm and then coat them with acrylic topcoat using flow/curtain coater (Appendix II).
5. Irradiate the topcoat with the low energy electron beam accelerator (Appendix IV).
6. Characterize the properties of the product such as hardness, glossiness, adhesion etc.

#### **Radiation Curing of Surface Coating Process**



## **Part II            PRACTICAL EXERCISES**

### **Coating and Characterization**

#### **i.        Substrates**

Glass plate and cement board (cemboard) will be used in this experiment.

#### **ii.      Coating Material**

Radiation curable coating material.

#### **iii.     Experimental Procedure**

1.    Clean glass plate with acetone thoroughly.
2.    Before coating, weigh the glass plate using Mettler balance.
3.    Coat the radiation curable coating material onto the surface of the glass plate by using a bar coater of size # 30.
4.    Then weigh the glass plate which has been coated with the coating material.
5.    Irradiate the sample by using the low energy electron beam accelerator at required dose.
6.    Carry out different types of tests on the above sample such as hardness, glossiness, adhesion properties.

**iv. Calculations**

**a. Absorbed Dose**

$$\text{Dose (kGy)} = \frac{K}{V \text{ (m/min)}} \times \frac{I \text{ (mA)}}{W \text{ (cm)}}$$

where,

K	=	Constant (169 at 200 kV)
I	=	Current
V	=	Conveyor Speed
W	=	Irradiation Width

**b. Pendulum Hardness (*Konig Principle*)**

$$\% \text{ Pendulum Hardness} = \frac{\text{No. of Oscillations (sample)}}{\text{No. of Oscillations (std)}} \times 100$$

**Limitation :** Minimum film thickness required at least 30  $\mu\text{m}$ , comparison is only possible with layers of equal thickness. Surface must be clean and even. Final installation place must be free of vibrations.

**c. Adhesion**

$$\% \text{ Adhesion} = \frac{\text{No. of Adhesion}}{\text{Total Adhesion}} \times 100$$

**2. Type of Substrate**

Substrate	:	
Weight of Substrate	:	g
Weight of Coated Substrate	:	g
Area of Coated Substrate	:	cm <sup>2</sup>
Coating Thickness	:	



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## REPORT

Date :

Name :

### Practical Exercise I

#### Coating and EB Curing by Curetron

#### 1. Low Energy Electron Beam Accelerator Curetron Model EBC-200-20-15

Beam Voltage	:	kV
Beam Current	:	mA
Oxygen Concentration	:	ppm
Conveyor Speed	:	m/min
Vacuum	:	Pa
Dose	:	kGy

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### Practical Exercise II

#### Testing of EB-treated Coatings

##### 1. Pendulum Hardness Test

Material	Oscillations			Average	% P.H.
Glass (std)					
Sample I					
Sample II					

##### 2. Gloss Test

Glossiness	I	II
Standard		
Sample I		
Sample II		

##### 3. Adhesion Test

Sample No.	No. of Adhesion	No. of Failure	% Adhesion
I			
II			

## Appendix I

## Procedure to use Automatic Reverse Roller Coater

Model RC - 13  
TANOUCHI IRON WORKS, Japan.

## APPLICATION

Base coating, sealing, and finish coating for the plain surface of Fancy plywoods, Blackboards, Table-tennis tables, Concrete panels, Particleboards, Cabinets, Furniture components, Plasterboards can be done speedily.

Also the machine can be used as a Glue Spreader.

## SPECIFICATION

Natural-side feeding speed: 10-40 m/min

Reverse-side feeding speed: 6-60 m/min

Effective thickness: 1-70  $\mu$ m

Roller size: Coating rubber roller;  $\varnothing$  190 x 470  $\mu$ m (Rubber lining)

Doctor roller ;  $\varnothing$  138 x 470  $\mu$ m (Chrome plated)

Reverse roller ;  $\varnothing$  214 x 470  $\mu$ m (Chrome plated)

Doctor roller ;  $\varnothing$  138 x 470  $\mu$ m (Rubber lining)

Backup roller ;  $\varnothing$  190 x 390  $\mu$ m (Chrome plated)

Effective width: Back-up roller 370  $\mu$ m

Required power: Motor for the coating roller drive; 0.4 kW Safety & Nonspark

Motor for the reverse roller drive; 0.75 kW Safety & Nonspark

Motor for the pump ; 0.4 kW Safety & Nonspark

Pass line: 850 mm

## CONSTRUCTION AND FUNCTION

Construction of the machine is as shown on the general drawing: It has two side-frames and they hold 4 rollers and 2 doctor rollers. The rollers are driven by special couplings through the gear box. The rollers rotate very smoothly and accurate coating operation can be done. The coating roller and the doctor roller have a different circumferential speed, and it is easy to control the coating amount.

## NOTICE FOR OPERATION

a) Please make sure to do the following before the operation

\* Lubrication

Machine oil for metals and sliding parts: 1~2 times/day

Grease for bearings and couplings: 1 time/month

Gear oil for gear box: 1 time/month

\* Adjustment of gap of the rollers

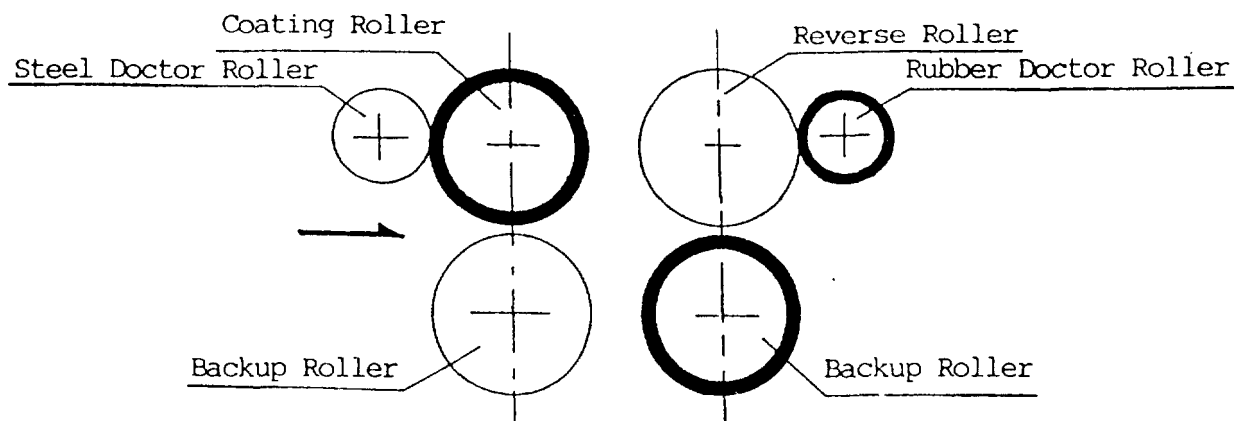
Doctor roller and Coating roller(Reverse roller)

Coating roller(Reverse roller) and Backup roller

b) Operation Start

Please check the rotation direction of the rollers when you start operation.

Refer to the following drawing and check if the board is fed as the direction of the arrow.



c) The great notice at the beginning of the operation: Do not let the doctor roller and the coating roller touch each other without paint, otherwise the rubber of the coating roller will be damaged.

\* Supply paint on the rollers when you start operation. Then adjust the coating amount. It is done by the adjusting(flower-shaped) handles.

\* After you supply paint on the rollers, please operate continuously.

After you stop the machine, paint on the rollers becomes solid. The solid paint will damage the rubber rollers at the next operation if you do not clean up fully the solid paint. The greatest care has to be taken if you use kinds of paint which solidify fast.

d) Solidifying Time of Paint

Please try to finish the operation within the solidifying time(The time is that the paint can be used with the machine, not the time of actual solidifying of the paint.). If it passed the solidifying time, the solidified paint would damage the rubber rollers. Try to keep supplying the paint.

NOTICE AT THE END OF THE OPERATION

- a) Please be sure to clean up the machine very carefully. Take all the paint off of the rollers with cleaning thinner. If this is not done enough, wear of the rollers will be much faster.
- b) Please supply oil and grease to the machine. Bush metals and sliding parts need the great care of oiling.

ADJUSTMENT FOR DIFFERENT THICKNESS OF BOARDS

- a) There is the adjusting handle at the left-side of the machine from the input-side. The handle is to adjust the gap between the rollers so that you can input boards with different thickness. The handle has a scale and an arrow.
- b) The gap between the coating roller and the buckup roller is to be 0.2~0.3 mm less than the thickness of the board which is to be coated.

If you set the gap too small, it would give the machine overload and might cause unexpected troubles.

ADJUSTMENT OF COATING AMOUNT

Adjust the amount depending on viscosity and character of the paint.

Adjustment can be done by the adjusting(flower-shaped) handles. Rotate the both handles at same amount to adjust the gap between the coating roller and the doctor roller.

## Procedure to use Flow/Curtain Coater

Model FL - S3F

IWATA AIR COMPRESSOR MFG CO LTD, Japan.

### Operation Adjustments

The following preparation procedure and checks are required before starting operation.

#### Preparation for Operation

- (1) Check to see that there are no unnecessary objects on the conveyer belt.
- (2) Check all joints and clamped parts of the paint circulating route to confirm that they are fixed securely.

#### Configuration

##### Main Components of Flow Coater

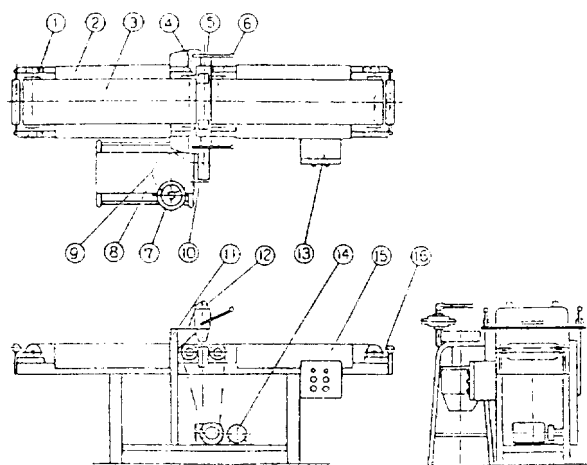


Fig. Block Diagram of Flow Coater

No.	Parts Designation	No.	Parts Designation
1	Conveyor roller	9	Paint container
2	Belt support plate	10	Paint receiver
3	Conveyor belt	11	Head stay
4	Fixed head	12	Head support bearing
5	Moving head adjusting shaft	13	Control panel
6	Clamp lever	14	Speed control unit for conveyor
7	Filter chamber set	15	Belt support stay
8	Pump unit	16	Slide base

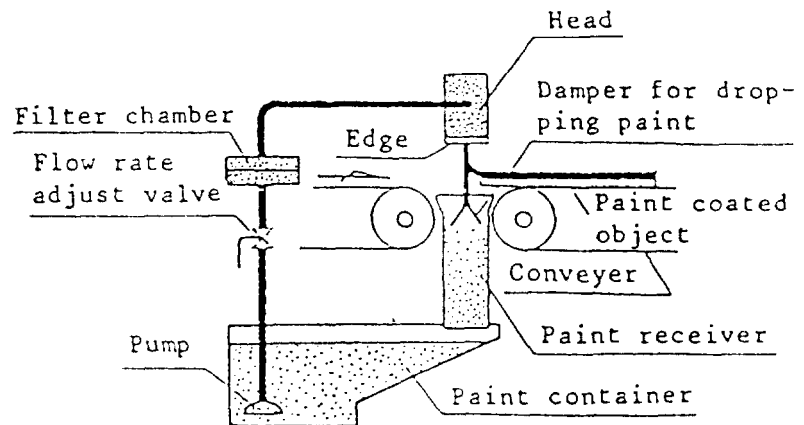
- (3) Adjust the slit width off the edge to an optional setting in the range of 0.3~1.0mm.

A slit width of 0.5~0.8mm is normally used.

- (4) Before charging of the paint in the Flow Coater, clean the paint circulating route (Pump unit, paint container, paint receiver, inside of head, and inside of filter chamber) with waste cloth. Then, circulate thinner through the route to it clean.

After draining the thinner used for cleaning, close the flow rate adjust valve and by-pass valve. (Turn valve to "Close" position.)

- (5) Pour the appropriately diluted paint into the paint container. Pour in the paint slowly along inclined part of the container so that no bubbles will form.



- (6) Replace the screw of the speed control dial.

Fig. Paint Circulating Route of Flow Coater

(Detailed explanation paper is attached to speed control unit.)

#### Operation Adjustment

Turn ON the power supply switch. Then, turn ON the conveyer switch.

Adjust the conveyer speed to 60~80m/min. and check to confirm that the belt is operating properly.

### (1) Tension of Conveyer Belt

Adjust the tension of the belt by turning the take-up bolts provide at both ends of the conveyer. Loosen

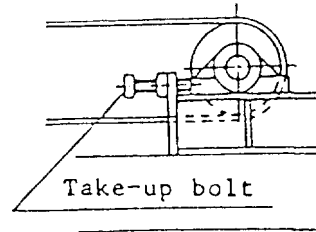


Fig. Take-up Bolt

the bearing bolt slightly, first, before

adjustment of the take-up bolts. The normal take-up range for the belt tension is 0.2~0.4%.

Do not tighten the belt too much because excessive tension may result in damage of the roller shaft. For adjustment of the belt tension, loosen the conveyer belt and mark the beginning and end points of a 1000mm section of the belt. Then, tighten the take-up bolts until the marked section becomes 1002~1004mm.

### (2) Adjustment to prevent Side-slip of Conveyer Belt

When there is side-slip of the conveyer belt during operation, adjust by means of the take-up bolts. Since the follower roller has a crown shape, it will act to correct any side-slip. Therefore, the side-slip can be stopped by only a slight adjustment of the take-up bolts.



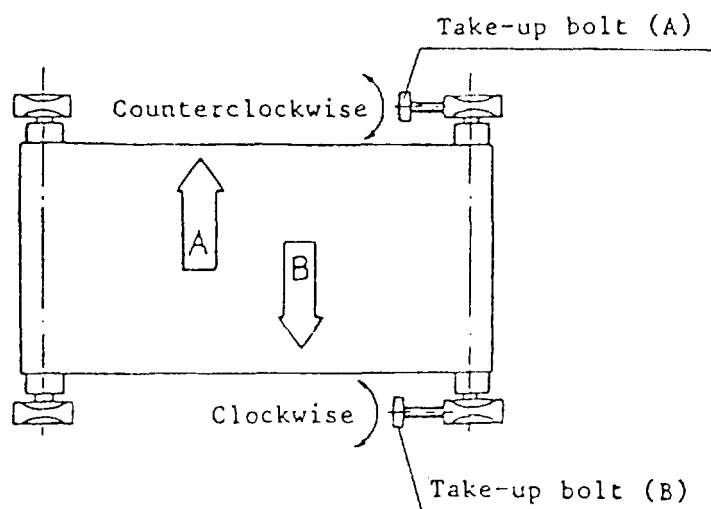


Fig. Top View of Conveyor

#### Adjustment of Conveyor Belt Side-slip

Adjust Method Correcting Direction	Take-up Bolts
To return belt in direction A.	Turn take-up bolt (A) counter-clockwise Or Turn take-up bolt (B) clockwise
To return belt in direction B.	Turn take-up bolt (A) clockwise Or Turn take-up bolt (B) counter-clockwise

### (3) Conveyor Speed Control

The conveyor speed can be increased or reduced by turning the conveyor speed control dial during operation. The conveyor speed corresponding to the scale reading of the dial is shown in the conversion table provided at the end of this manual. Do not turn the speed control dial when the conveyor is stopped in any case.

(4) Adjustment of Paint Supply Rate

Turn ON the pump drive switch, and gradually "Open" the flow rate adjust valve which was fully closed. (Cock scale 0 to 1, 2, 3 and 4.)

In case paint of ordinary viscosity is used, the curtain will become ununiform if the flow rate is too high.

A uniform film curtain can be obtained by gradually closing the valve to obtain the proper flow rate in such a case.

Although the flow rate is increased in some cases according to the required coating conditions, the best curtain condition can be obtained at the flow rate just before the point where the curtain will break in case of general paint coating operation.

As a scale is provided on the flow rate adjust valve, the scale setting for the optimum condition should be kept on record to facilitate readjustment when a curtain break occurs.

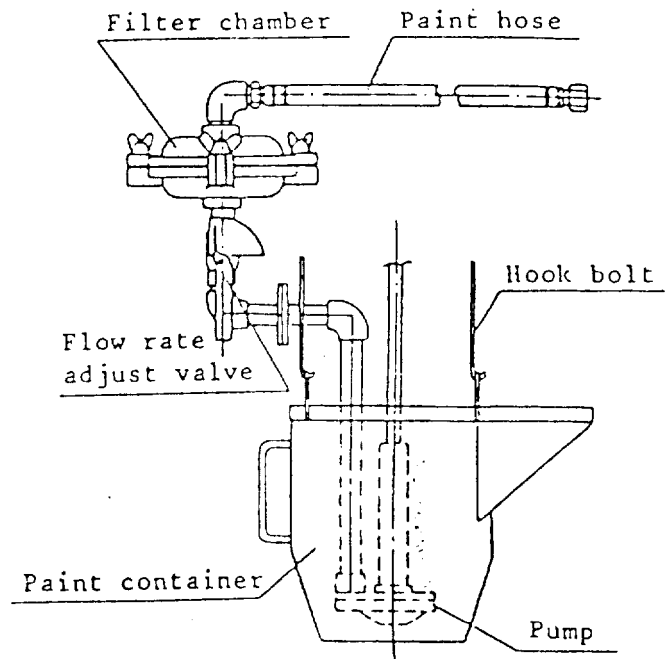


Fig. Pump Section

This completes the adjustment procedure. The object to be coated can now be placed on the conveyer and be passed through for coating. Adjustment of the coating volume will be required if the coating thickness.

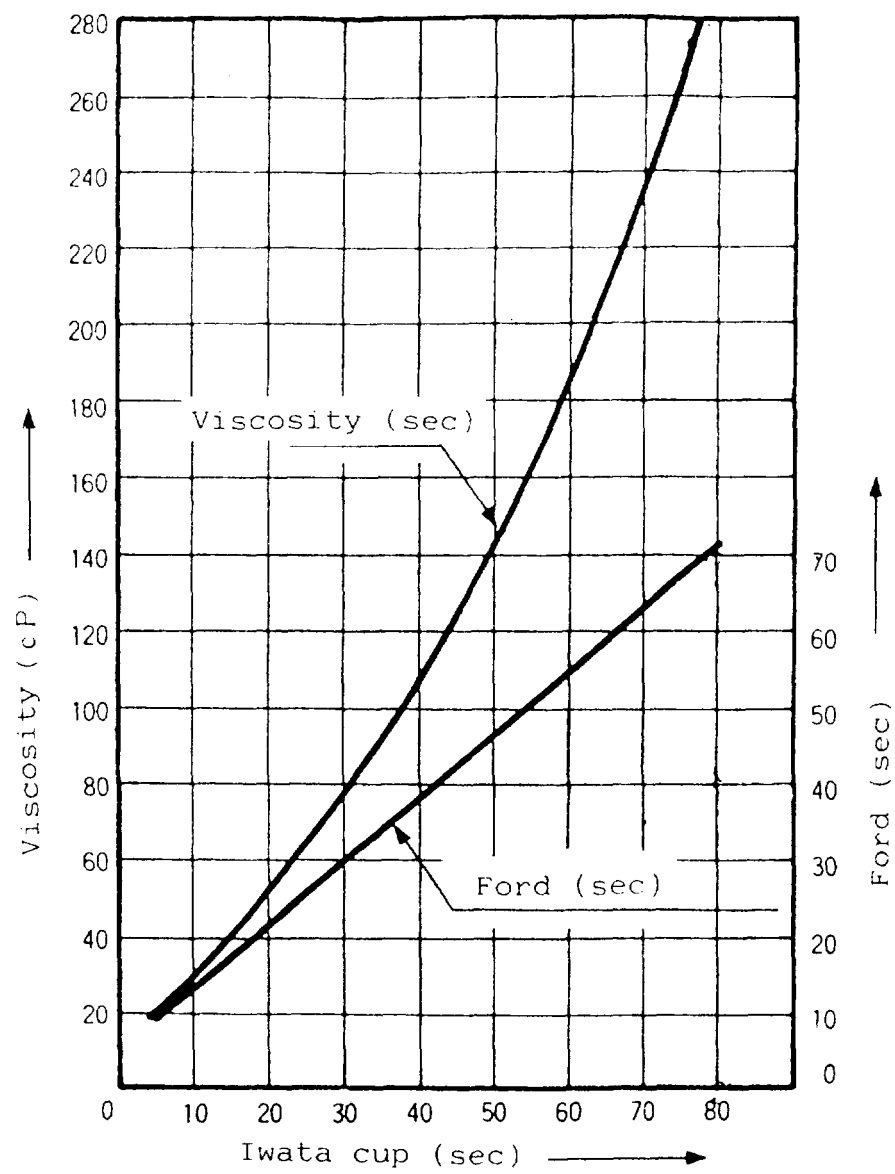
#### Coating Volume Adjustment

The adjustment items and criteria to obtain the proper coating volume are given in the following table.

Sequence	Adjustment Item	Adjustment Criteria
1	Paint viscosity adjustment.	Adjust to proper viscosity for required coating conditions.
2	Slit width adjustment.	Determine required slit width according to quality and viscosity of paint to be used.
3	Flow rate adjust valve.	Throttle the value until film curtain break will occur.
4	Conveyer speed adjustment	Adjust to speed setting where desired coating thickness is obtained.

#### (1) Paint Viscosity Adjustment

Adjust to lower viscosity when a thinner coating is desired. And adjust to a higher viscosity when a thicker coating is desired.



(2) Slit Width (Clearance between 2 edges)

Adjustment the slit width can be adjusted by loosening the head clamp lever and turning the stoppers on the left and right sides of the head. The slit will become wider when the stopper is turned clockwise, and will

become more narrow when it is turned counterclockwise.

A uniform film curtain can be obtained by gradually closing the valve to obtain the proper flow rate in such a case.

Although the flow rate is increased in some cases according to the required become more narrow when it is turned counterclockwise.

One scale graduation of the stopper corresponds to 0.1mm

adjustment and one turn corresponds to 1.0mm adjustment of the slit width.

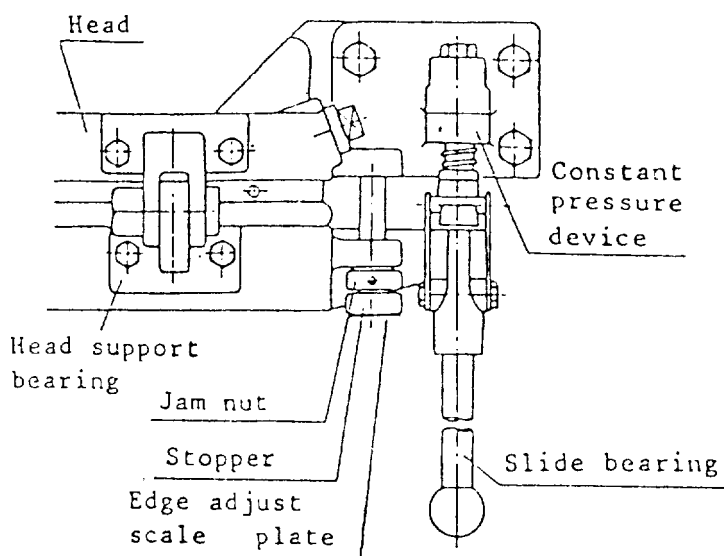


Fig. Head Section

Scale Reading and Slit Opening (For reference)

Scale	Slit Opening (Width)
0	0 m/m
2	0.27
4	0.53
6	0.80
8	1.06
10	1.33

However, since this scale is only a criterion for adjustment, use the clearance gauge for final check of the adjustment. Set the head clamp lever in its original position and use the clearance gauge to check whether the width is uniform along the entire slit.

After the desired slit adjustment is obtained, tighten the jam nut securely so that the adjustment will not be deranged.

#### Slit Width Adjustment Criteria

A slit width of 0.5~0.8mm is normally used.

Good results cannot be obtained with a slit width of less than 0.5mm or more than 0.8mm, except in the case where a special paint is used.

The slit width could not be adjusted with the wrong belief that a thicker coating can be obtained by a wider slit or that a thinner coating can be obtained by a more narrow slit.

The paint curtain will be more stable when the slit width is adjusted as wide as possible within the range where the film curtain is formed.

#### (3) Adjustment of Paint Supply Rate

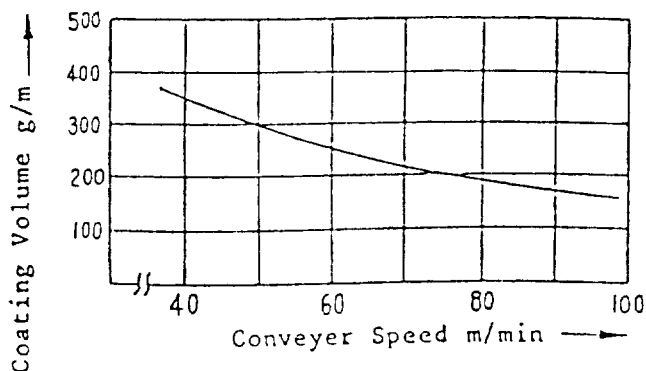
The supply rate is adjusted by the flow rate adjust valve. After the slit width has been determined, supply the appropriate volume of paint for the slit width to the head.

## (4) Conveyor Speed Adjustment

After an ideal film curtain is formed by the procedure explained in the foregoing 3 items, produce the desired coating thickness by the conveyor speed adjustment which is the final adjustment.

Although the coating volume will be reduced when the conveyor speed is raised, high speed operation will cause poor stability of the curtain and will increase the possibility of the curtain being cut by the wind pressure when the object to be coated is thrust into the curtain. Therefore, coating operation with the conveyor speed set as low as possible will be ideal.

Refer to the following graph. (This data is only one example.)



(Condition)

Paint: Lacquer  
 Viscosity: 30 sec./I.H.S. cup  
 Slit: 0.6mm  
 Valve opening: 4.0

## (5) Adjustment Methods and Effectiveness

Adjustment item	Degree of coating volume change	Change of curtain condition	Fine adjustment
Adjustment by slit width	Small	Change occurs	Difficult
Adjustment by flow rate adjust valve	Large	Change occurs	Rather difficult
Adjustment by conveyer speed	Medium	No change	Easy

## Stop of Operation

- (1) Turn OFF the conveyer switch after the coating operation is completed.
- (2) Turn OFF the pump switch after the coating operation is completed.
- (3) Place the belt cover on the belt under the head. This belt cover is also put on the belt during adjustment of the paint curtain to prevent soiling of the belt.



## DESCRIPTION and TECHNICAL DATA of IST-UV IRRADIATOR

### General Outline

The IST polymerization unit is designed to cure printing inks, varnishes as well as plastic coatings. The polymerization is obtained by intensive, pulsed UV radiation, inducing the curing in a very short period. The polymerization is a chemical process compared to physical curing in the presence of solvents.

Coating materials on the base of the following binding agents are normally curable:

Sensitized acrylic resins, unsaturated polyester, linked polyurethanes as well as synthetic resins in sensitized form, which are curable by energy absorption on account of their chemical structure. These systems are curing without any solvents.

Coating materials which are exclusively curable by evaporation of the solvent are generally not reacting.

### Carrier:

The carrier has no direct influence on the curing and is determined by the field of application. The adhesion (in the case of plastic material) is very often affected by the carrier.

### ATTENTION

A highly intensive, short-wave ultra-violet radiation is produced in the unit (similar to the radiation of sun at high altitudes). The skin and particularly the eyes must not be exposed to this radiation. Therefore look into the unit only in exceptional cases and with suitable goggles.

In operating condition the unit is so shielded that radiation detrimental to health cannot get out.

### Ozone content

A sufficient quantity of air is supplied to each lamp so that the ozone content during operation does not exceed the maximum admissible value of 0,1 ppm. The measurements indicated a value inferior to 0,05 ppm (no reaction at measuring tube).

### Measured by means of:

Dräger tube 0,05/a in the exhaust air stream (measuring range 0,05 to 1,4 ppm).

### Remark:

A much lower quantity of ozone can be detected by smelling, however this concentration can hardly be measured.

An adjustable radiation shielding is fitted at the intake and discharge side of the radiation housing. It must be adjusted to the thickness of the passing material to ensure an optimum protection against radiation.

**POOR QUALITY  
ORIGINAL**

Technical Data

Equipment specification Type	IST-UV Dryer Minicure U 200 - M - 1 - Tr
Finish	RAL 9001 "white-cream"
Operating voltage	3 x 415 V, 50 cycles
Control voltage	240 V, 50 cycles
Connected load	approx. 4 kW
Connected load of lamps	2 kW

## Dimensions:

Length	1.000 mm
Height	850 mm + 80 mm vertical adjustment
Overall width	1.045 mm
2 Reflectors type	288 mm
1 IST-Lamp type MC	200 mm (Minicure)
1 Thermostat	175°C
1 Thermostat	50°C

Switch Cabinet

## Dimensions:

Length	600 mm
Height	380 mm
Depth	210 mm

1 Main switch

1 Amperemeter

1 Service hour meter

Various operation and control instruments.

Conveyor

Length	1.000 mm
width	436 mm
Working width approx.	440 mm
Speed	0 - 40 m/min. infinitely adjustable
Roller diameter	120 mm
Distance between rollers	860 +/- 10 mm belt tensioning device
1 Conveyor belt VA	
Length	2.080 mm
Width	240 + 2 x 14 mm gear edge
Mesh length	5 x 48 mm

1 Direct current gear motor type	SN 9 FG; 0,25 kW
1 Thyristor regulator type	ED 20 00 11
1 Analog display	96 x 96
1 Retardation coil	3 A
1 Nominal value potentiometer	

Fans

1 Exhaust air fan type	DNG 3 - 9,8 S 15
motor capacity	N = 0,24 kW
speed	n = 2800 rpm
air volume	V = 180 m <sup>3</sup> /h for 430 Pa

Exhaust air

Total quantity of exhaust air	200 m <sup>3</sup> /h
Section of exhaust ducting to be placed by customer	1 x NW 100
Max. length of exhaust ducting	approx. 8 - 10 m
0,75 m Exhaust air hose	NW 100

## Description of Construction and Operation

### Survey

The coated substrate is conveyed through the radiation area by a conveyor and the ink or varnish is cured in a very short time. After leaving the radiation area the material may be withdrawn from the conveyor.

### Radiation Unit

The radiation unit consists of a stable aluminium construction and may be taken off for cleaning and maintenance works. The lamp and reflector are mounted in an interior housing and are cooled by vacuum intake air which ensures at the same time that the lamps and reflectors are not excessively heated up and that the produced ozone vapors are directly extracted. Simultaneously the temperature at the object is reduced. The exhaust air fan is mounted at the radiation housing. The radiation housing is vertically adjustable by 4 x 20 mm by means of pads of isolating material.

### Conveyor

The conveyor consists of a stable steel tube frame with sheeting. The drive is arranged at the discharge side which brings about a pulling transport motion. The unit is driven via a simplex roller chain with chain tensioning device.

The fine steel belt is led over a drive and guide roller. The rollers are grooved to guide the meshes of the belt and avoid its lateral shifting. The rollers are fixed at the conveyor frame by each two swing bearings and belt tensioning device. A guard plate is provided at the intake and discharge side.

### Belt tensioning device

To operate the belt tensioning device the guard plates at the intake and discharge side must be screwed off first.

The lateral fixing screws are untwisted by one or two turns (4 off for each seating). The rollers are now adjustable in both directions by means of the front adjusting screws. Attention must be paid that the rollers are adjusted at right angles to the conveyor and in parallel to each other. The fixing screws are tightened and the guard plates screwed on.

### Electric Switch and Control Unit

This unit is fixed laterally at the conveyor and comprises the complete control and operation elements.

The power supply devices necessary for each lamp are lodged in the under-structure of the vacuum conveyor.

Safety Precautions

General Remarks

1. Efficient ventilation of the IST lamp and object.
2. Caution boards fixed at the unit.
3. Ozone content: A sufficient quantity of air is supplied to each lamp to secure that the maximum admissible concentration is not exceeded during operation.
4. Cooling down of the unit by follow-up adjustment.
5. The functioning of the fans is controlled by bimetal overload relays and failures are indicated.
6. In case of excessive temperature of the lamps a built-in maximum thermostat automatically cuts the circuit thus protecting the lamps and reflectors. The pilot lamp "excessive temperature of lamp" lights up at the control cabinet.

P93

**Low Energy Electron Beam Accelerator (Curetron)  
Model EBC-200-20-15**

**Introduction**

This equipment produces electron beam which is used for research and development of paints and resins curing by direct injection of energy.

**Features**

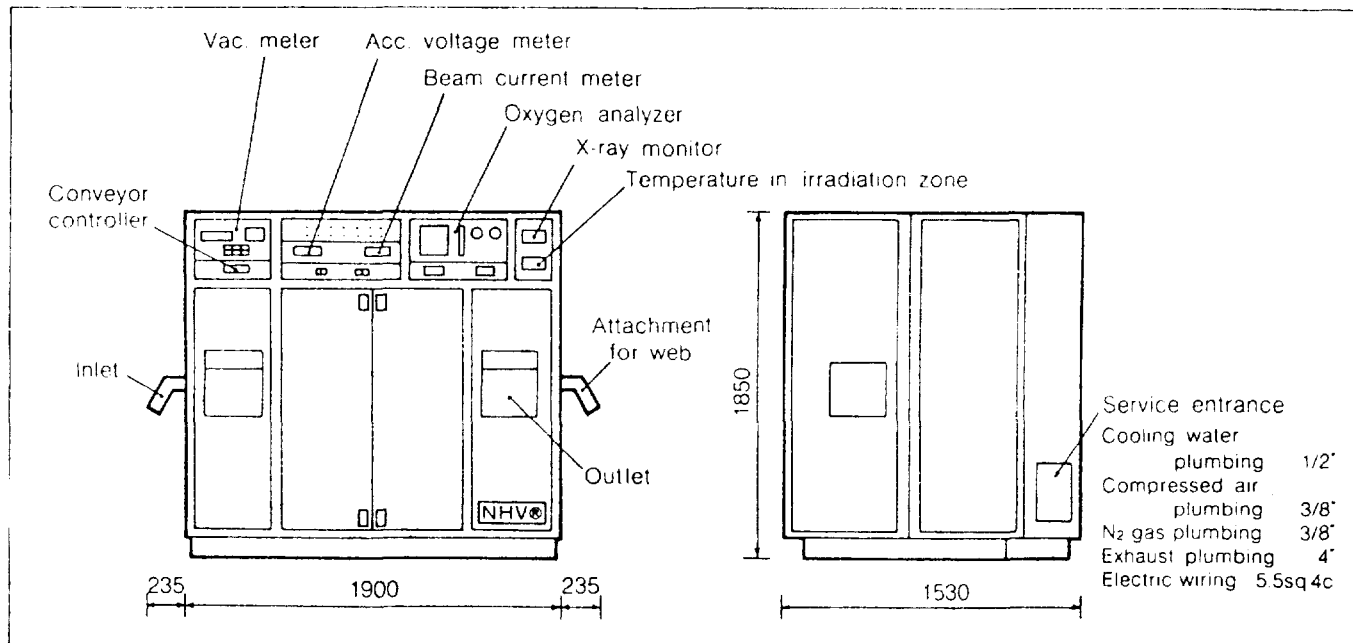
1. Compact design - not requesting much installation space.
2. The equipment is completely provided with an electron beam accelerator, a conveyor system, an inert gas chamber, an oxygen analyzer and a safe X-ray shielding.
3. Easily operated by only pressing a few buttons.
4. Continuous web irradiation can be achieved by fitting inlet and outlet attachment.

**Ratings and Utility Requirement**

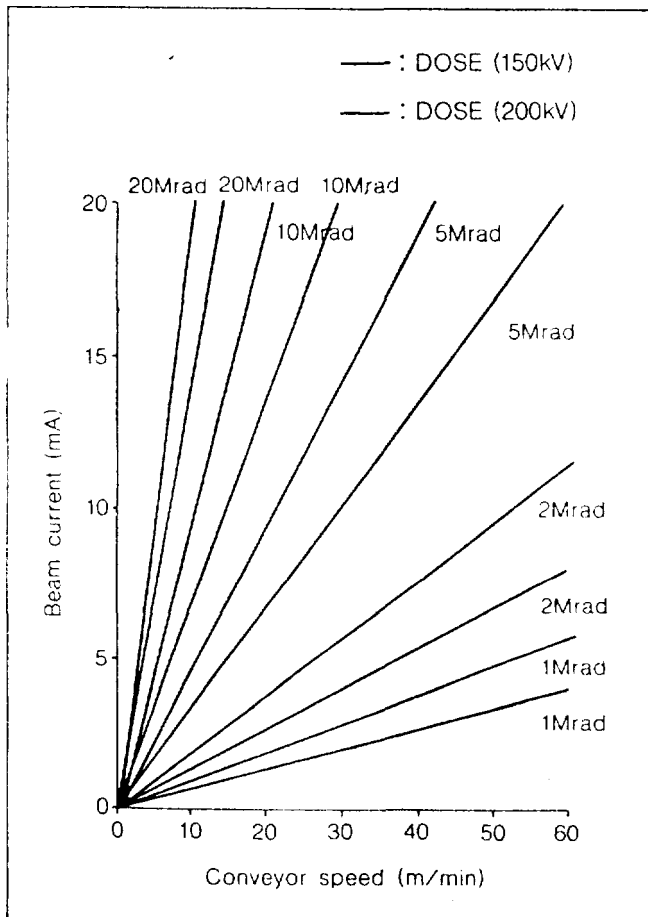
Accelerator voltage	:	150 - 200 kV	
Beam current	:	20 mA	
Irradiation width	:	15 cm	
Conveyor speed	:	3 m/min - 60 m/min	
Sample size	:	15 cm x 15 cm	
Dose uniformity	:	± 10 %	
Electric power	:	3 phase 200V 50/60 Hz	10 kVA
Cooling water	:	13 liters/min	
Nitrogen gas	:	2 - 3 m <sup>3</sup> /hr	

# AREA BEAM Curetron®

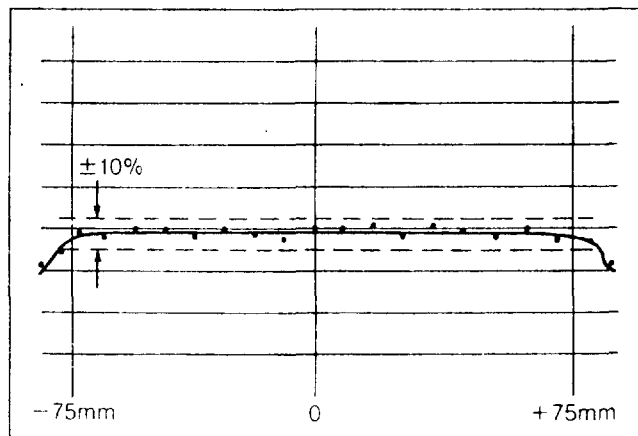
## ■ OUTLINE



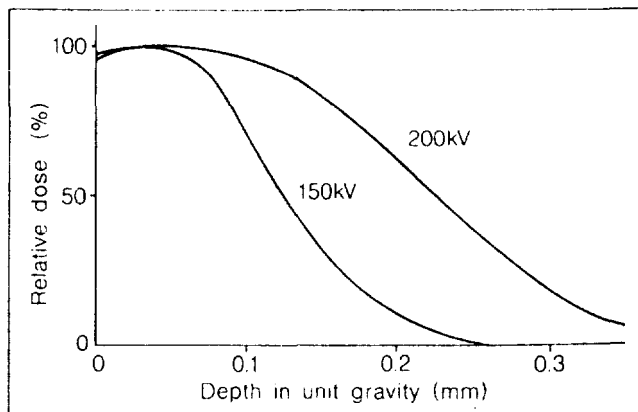
## ■ DOSE



## ■ DOSE UNIFORMITY



## ■ PENETRATION



**#8**

**DEVELOPMENT OF LENRA  
AND ITS APPLICATION**





## Development of Radiation-curable Resin based on Natural Rubber\*\*

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### Abstract

A new radiation curable resin based on natural rubber has been developed. The resin was based on the reaction between low molecular weight epoxidised natural rubber and acrylic acid. When formulated with reactive monomers and photoinitiator, it solidified upon irradiation with UV light. The resin may find applications in coating for cellulosic-based substrates and pressure-sensitive adhesive.

### INTRODUCTION

Malaysia is one of the world's biggest producer of natural rubber [1]. There has always been great interest in the modifications of natural rubber as a means of improving the properties of it as well as to develop new materials. Various products such as chlorinated [2], hydrochlorinated [3], epoxidised [4,5], grafted [6] and liquid [7,8] natural rubber have been reported.

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\* A lecture prepared for the Workshop on Surface Finishing by Radiation Curing Technology, being held at Kompleks PUSPATI, Bangi, 8-9 September, 1993.

Natural rubber consists of long chain molecules, each with repeated unsaturated double bonds at every five carbon atoms [9]. The unsaturated double bond is the functional group of the rubber molecule which can be reacted to form new materials [10,11,12,13].

Recently, epoxidised natural rubber (ENR) has been commercially produced by a local company. Its development was originally carried out at MRPRA in late 1970s before shifting to RRIM for scale-up studies in 1980s.

The company produces two grades of ENR i.e. ENR-25 (epoxide content of 25 mole %) and ENR-50 (epoxide content of 50 mole %). Both products are in the form of solid with estimated weight average molecular weight,  $\bar{M}_w$ , of over 300,000. To make the products more useful, methods have been established by RRIM and UKM to lower the molecular weight of ENR. The availability of low molecular weight ENR enables studies to be carried out to prepare radiation-curable resin based on natural rubber [14]. For this country, besides palm oil [15], rubber holds a promising potential to be the next renewable raw material substitutes for industrial chemicals especially radiation curable resins.

The majority of the radiation curable resins currently commercially available are products of non-renewable petroleum-based synthetic raw materials. This report briefly describes basic formulations necessary for a newly developed low molecular weight epoxidised natural rubber acrylate (ENRA) to be used as UV curable resin.

EXPERIMENTAL

**Materials:** Low molecular weight epoxidised natural rubber (ENR) with various molecular weights were supplied by courtesies of Dr. Ibrahim Abdullah, Chemistry Department, UKM and Dr. Sidek Dulngali, RRIM. The former were prepared from ENR supplied by Guthrie Corp. Berhad, Chemara, Negeri Sembilan. For this formulation work the resin was prepared from low molecular weight ENR-25 supplied by UKM with  $\bar{M}_w$  and  $\bar{M}_n$  of 147,000 and 39,000 respectively. Acrylic acid which contained 200 ppm hydroquinone monomethyl ether (Merck, 99%) and toluene (Merck, 99%) were used as received. Triethylamine (Merck, 99%), p-methoxyphenol (Aldrich, 98%) were used as catalyst and inhibitor respectively.

Two monomers i.e. tripropylene glycol diacrylate (TPGDA) and trimethylol propane triacrylate (TMPTA); three photoinitiators and photoactivator i.e. benzophenone (BP) and 1-benzoylcyclohexanol (Irgacure 184) and N-methyl diethanol amine (N-MDEOA) were used for formulation.

**Synthesis Apparatus:** The apparatus involved were 1 L round-bottomed flask equipped with a condenser, a thermometer, a mechanical stirrer and a dropping funnel.

**Synthesis Procedure:** The low molecular weight ENR sample was first dissolved in toluene in a reaction flask to a required percentage (v/w). 0.5 or 1.0% p-methoxyphenol was mixed with 1.0% triethylamine before adding to the reactants.

The mixture was stirred and heated up to 100°C. Then acrylic acid was added dropwise. The temperature was maintained throughout the reaction. The progress of the reaction was monitored by measuring the acid number of the mixture.

**Formulation Procedure:** When the acrylation reaction terminated at certain acid number, toluene was replaced with a reactive monomer i.e. TPGDA or TMPTA by rotary evaporation. Then the complete formulations were prepared by adding other relevant chemicals as indicated in Tables 3 to 5. Their viscosities were measured using a Well-Brookfield DV II viscometer model RVTDV-II CP with a spindle # CP-41. The formulations were later coated onto 10cm by 10cm glass plates using a drawdown bar coater (RDS 30, Webster) to give 30  $\mu$ m thickness and then cured with a 20 cm width IST Minicure 200 machine fitted with a UV lamp of 80 W/cm and an operating current at 7.0 amp. When cured, the samples were subjected to a number of simple instantaneous qualitative tests i.e. tackiness and finger marring. The hardness of the cured films was measured with a Byk Labotron Pendulum Hardness Tester (Konig method). Gel fraction was measured by soxhlet extraction method.

**Molecular Weight Measurement:** It was done using Waters GPC 200 machine equipped with four prepacked Styragel columns. Tetrahydrofuran and polystyrene were used as a solvent and standard respectively.

**IR Measurement:** The functional groups of ENR and its acrylated derivative were measured with a Nicolet 60SX Fourier Transform

infra-red spectrophotometer.

Chemical Resistance Tests or Chemical Spot Tests: The tests were carried out using chemicals such as 5% acetic acid, 50% ethanol, 1% sodium carbonate, 10% sulphuric acid and 10% sodium hydroxide.

### RESULTS AND DISCUSSION

Acrylation Reaction: Theoretically speaking, almost any epoxide group can be acrylated given the right conditions [16]. But in case of ENR there are several conditions to be observed in order to minimise the risks of gelation during synthesis. Table 1 summaries the synthesis data as a result of these various conditions.

In an ordinary natural rubber, there is only one functional group i.e. the C=C double bond. When the rubber is epoxidised in correct conditions, only epoxide groups are formed [4]. In other conditions, complex products containing hydroxyl and ring-opened structures are also obtained [17,18]. Depolymerization of the rubber shows the formation of hydroxyl group [19]. In the case of acrylation process, some changes in IR peaks are expected such as new peaks attributable to acrylate group and diminishing of epoxide peaks [15].

But only three functional groups are of importance to us i.e. epoxide group (-CHOCH-) in ENR and the other two are acrylate ( $\text{CH}_2=\text{CH}-\text{COO}-$ ) and hydroxyl (-OH) groups in low molecular weight

ENRA. These functional groups are clearly shown by IR spectroscopic technique in Table 2. When acrylation process takes place the epoxide peaks ( $1250$  and  $881\text{ cm}^{-1}$ ) almost disappeared and new peaks ( $1664$ ,  $1638$ ,  $986$  and  $809\text{ cm}^{-1}$ ) representing acrylate group appeared.

The reaction is illustrated by the equation below;

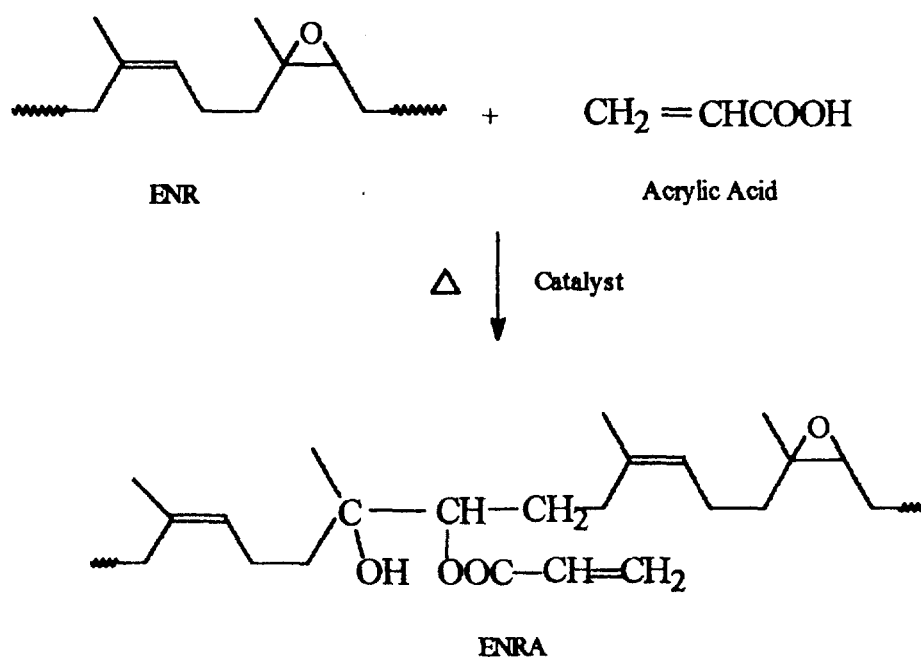


Table 2(c) shows the IR data of cured low molecular weight ENRA film. From this table it can be observed that the peaks attributable to the acrylate double bond have been used up. Upon irradiation in the presence of benzophenone, low molecular weight ENRA is capable of undergoing radical polymerization through its double bond from its newly attached acrylate group to form crosslinked network which is the basis of the radiation curing of

surface coatings.

#### UV Curing of Low Molecular Weight ENRA:

In these formulation studies, di- and trifunctional monomers were used i.e. TPGDA and TMPTA, respectively. They act as solvent and crosslinker.

Table 3 shows the effects of a single photoinitiator, BP and a combination of BP and a synergist, N-MDEOA on cured properties of low molecular weight ENRA using TPGDA as a reactive monomer.

From Table 3, it clearly indicates that the cure rate increases as the N-MDEOA content increases but at the expense of the gel fraction and the pendulum hardness. It is believed that the ability of N-MDEOA in enhancing the initiation reaction lies with its hydroxyl group which provides a source of readily extractable hydrogen [20]. The addition of N-MDEOA to BP repressed oxygen inhibition and enabled the latter to perform as effectively in air as in nitrogen [21]. On the other hand, single BP imparts higher gel fraction and pendulum hardness but the cure rate is slower as demonstrated by formulation A1.

With the addition of trifunctional acrylate i.e. TMPTA, the hardness and the cure rate were not improved as normally the case. Instead, the data in Table 4 indicate that their physical properties have been somewhat affected by the addition of TMPTA.

Table 5 illustrates the effects of Irgacure 184 and BP. The combination of both is commercially available as Irgacure 500

which is used for reducing an air inhibition effect in the absent of amines [22]. As shown by formulation B1 in Table 5, the cure rate increases more than double that of A1 - the one without Irgacure 184. At the same time, Irgacure 184 also reduces the negative effect of TMPTA on the pendulum hardness as shown by formulation B4. In short, with Irgacure 184, the physical properties of the coatings are found to be better than the formulations with the combination of BP and N-MDEOA.

The formulations also show good chemical resistance to a number of chemicals tested.

In other development [27], we used rubberwood as a substrate to be coated with natural rubber-based formulation. The formulation and performance of the coating film are given in Tables 6 and 7, respectively. The results show that its performance is comparable to commercial resins.

### CONCLUSIONS

Since the preparation of new resin requires the use of solvent, the amount of solvent needed depends on the molecular weight of the starting material, ENR. The lower the molecular weight, the higher the concentration of ENR solution possible.

From formulation studies, several conclusions can be made such as;

- i. Diacrylate monomer, TPGDA delivers better performance than



triacrylate, TMPTA.

- ii. Irgacure 184 is better photoinitiator for the new resin.
- iii. New resin based formulations show good chemical resistances.
- iv. Their gel fraction is readily acceptable.

This new resin has potential to find applications in;

- a. Coatings for wood-based substrates (e.g. fillers and top-coats), paper and board, and
- b. Pressure-sensitive adhesives.

#### ACKNOWLEDGEMENTS

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Last but not least, we acknowledge financial support from the IRPA Code # 2-06-05-012.

\* More detailed reports are being published in Nucl. Sci. J. Malaysia, Vol. 11(1), 1993.

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Table 1 : Some data of the preparation of low molecular weight ENRA.

No.	Sample	Source	$\overline{M}_w$	$\overline{M}_n$	SC, %	OOO, %	Epoxide: AA ratio	Cat. %	Inh. %	Reaction time (hr)	Initial AN	Final AN	% AN reduction
1.	LENR-25	UKM	380,000	82,000	27.6	5.04	1:1.2	1.0	1.0	61	166	64.0	61.4
2.	LENR-25	UKM	380,000	82,000	27.6	5.04	1:1.2	1.0	1.0	116	166	55.4	66.6
3.	LENR-50	UKM	232,000	62,000	21.9	5.26	1:1.2	1.0	1.0	7	172	153.9	10.5
4.	LENR-50	UKM	232,000	62,000	21.9	5.26	1:1.2	1.0	1.0	15	172	141.5	17.7
5.	LENR-50	UKM	232,000	62,000	21.9	5.26	1:1.2	1.0	1.0	41.5	172	103.5	39.8
6.	LENR-50	RRIM	19,478	5,847	50	7.43	1:0.2	1.0	1.0	20	65.1	49.4	24.1
7.	LENR-50	RRIM	19,478	5,847	50	7.43	1:0.1	1.0	0.5	13	24	14.9	37.9
8.	LENR-50	RRIM	19,478	5,847	50	7.43	1:0.1	1.0	0.5	11	25	17.2	31.2
9.	LENR-50	RRIM	19,478	5,847	50	7.43	1:0.2	1.0	0.5	28	72	49.0	31.9

Notes :-  
 SC = Solution Concentration  
 OOC = Oxirane Oxygen Content (before reaction)  
 AN = Acid Number  
 Cat = Catalyst  
 Inh = Inhibitor  
 AA = Acrylic Acid

Table 2 : IR data of low molecular weight ENR-25, low molecular weight ENRA-25 and cured low molecular weight ENRA-25.

$\nu$ cm <sup>-1</sup>	Low molecular weight ENR-25 Assignment (a)	
3453.7	-OH	stretch.
2962.5	-CH	stretch.
2926.7	-CH	stretch.
2857.6	-CH	stretch.
1726	C=O	from lactone (?)
1664.8	C=C	stretch. of cis-1,4 [23]
1450	-CH <sub>2</sub> -	deform. mode [23,24]
1377.5	-CH <sub>3</sub> -	deform. mode [17]
1322.3		
1250.4	C-O	stretch. from epoxy [4]
1133.2	C-H	in-plane bend [17]
1081.4	C-O	stretch. from cyclic ether [17]
1036.0		
872.2	C-O	stretch. from epoxy [4]
836.9	C=C	vibration of cis-struct. [23,25]
741.8	C=C	vibration of cis-struct. [23]

.../more

$\nu \text{ cm}^{-1}$	Low molecular weight ENRA-25 (washed) Assignment (b)		$\nu \text{ cm}^{-1}$	Cured low molecular weight ENRA-25 Assignment (c)	
3476.9	-OH	(free) stretch.	3467 (broad)	-OH	(free) stretch.
2962.8	-CH	stretch.	2962.6	-CH	stretch.
2926.7	-CH	stretch.	2930.6	-CH	stretch.
2856.7	-CH	stretch.	2859	-CH	stretch.
2728.2	-OH	(bonded) stretch.			
			2360.7	CO <sub>2</sub>	peaks
			2340.8	CO <sub>2</sub>	peaks
1726	C=O		1732.3	C=O	
1664.2	}	external double bond and C=C of cis-1,4 [23]	1668.2	}	reduced double bond peaks
1637.6			1654		
1450.0	-CH <sub>2</sub> -		1450.5	-CH <sub>2</sub> -	
1404.3	C=C	external double bond (exomethylene group, scissoring =CH <sub>2</sub> )			
1376.9	CH <sub>3</sub> -		1377	CH <sub>3</sub> -	
1295.7			1316		
1269.7	}	$\begin{array}{c} \text{O} \\ \parallel \\ -\text{C}-\text{O} \end{array}$ stretch.	1255.4	C-O	stretch. from epoxy (reduced)
1192.2			1165.5	$\begin{array}{c} \text{O} \\ \parallel \\ -\text{C}-\text{O} \end{array}$	stretch.
1062.1	C-O	stretch. from cyclic ether (reduced)	1072.6 1029.7		cyclic ether (?)
985.5		CH <sub>2</sub> =CHR out of plane bending			
880.7	C-O	stretch. from epoxy (reduced), [4]	886.3	C-O	stretch. from epoxy
836	C=C	vibration of cis-struct.	837	C=C	vibration of cis-struct.
809.1	C=C	acrylic double bond [26]	745.4	C=C	vibration of cis-struct.

**Table 3 : Formulations of low molecular weight ENRA-25: Synergistic effects of N-methyl diethanol amine (N-MDEOA) with BP.**

FORMULATIONS	A1	A2	A3	A4	A5
OLIGOMER - Low molecular weight ENRA, %	20	20	20	20	20
MONOMER - TPGDA, %	75	75	75	75	75
- TMPTA, %	0	0	0	0	0
PHOTOINIATOR, % Benzophenone	5	4	3	2	1
N-methyl diethanol amine	0	1	2	3	4
VISCOSITY 25°C (cps)	1100	1100	1100	1100	1100
CURRENT, mA	7.0	7.0	7.0	7.0	7.0
CONVEYOR SPEED, m/min	2	2	2	2	2
NO. OF PASSES TO CURE (to a tack-free finish)	3X	2X	1X	1X	1X
PENDULUM HARDNESS, %	48.8	32.9	31.1	31.7	26.2
GEL FRACTION, %	95.2	92.3	94.5	94.0	90.7
CHEMICAL TEST (Yes/No)*					
Acetic acid, 5%	Y	Y	Y	Y	Y
Ethanol, 50%	Y	Y	Y	Y	Y
Sodium Carbonate, 1%	Y	Y	Y	Y	Y
Sulphuric Acid, 10%	Y	Y	Y	Y	Y
Sodium Hydroxide, 10%	Y	Y	Y	Y	Y

\* Yes means the spot is not affected and vice versa.

**Table 4 : Formulations of low molecular weight ENRA-25: The effects of TMPTA.**

FORMULATIONS	A6	A7	A8	A9	A10
OLIGOMER -					
Low molecular weight ENRA, %	20	20	20	20	20
MONOMER - TPGDA, %	65	65	65	65	65
- TMPTA, %	10	10	10	10	10
PHOTOINITIATOR, %					
Benzophenone	5	4	3	2	1
N-methyl diethanol amine	0	1	2	3	4
VISCOSITY 25°C (cps)	1040	1040	1040	1040	1040
CURRENT, mA	7.0	7.0	7.0	7.0	7.0
CONVEYOR SPEED, m/min	2	2	2	2	2
NO. OF PASSES TO CURE (to a tack-free finish)	5X	3X	2X	1X	2X
PENDULUM HARDNESS, %	45.5	24.7	31.7	26.8	24.4
GEL FRACTION, %	94.6	94.6	93.6	93.5	91.5
CHEMICAL TEST (Yes/No)*					
Acetic acid, 5%	Y	Y	Y	Y	Y
Ethanol, 50%	Y	Y	Y	Y	Y
Sodium Carbonate, 1%	Y	Y	Y	Y	Y
Sulphuric Acid, 10%	Y	Y	Y	Y	Y
Sodium Hydroxide, 10%	Y	Y	Y	Y	Y

\* Yes means the spot is not affected and vice versa.



Table 5 : Formulation of low molecular weight ENRA-25 : The effects of Irgacure 184 and BP.

FORMULATIONS	B1	B2	B3	B4
OLIGOMER -				
Low molecular weight ENRA, %	20	20	20	20
MONOMER - TPGDA, %	75	75	65	65
- TMPTA, %	0	0	10	10
PHOTOINITIATOR, %				
Benzophenone	2.5	0	2.5	0
Irgacure 184	2.5	5	2.5	5
VISCOSITY 25°C (cps)	940	940	1000 (est.)	1000 (est.)
CURRENT, mA	7.0	7.0	7.0	7.0
CONVEYOR SPEED, m/min	4	4	4	4
NO. OF PASSES TO CURE (to a tack-free finish)	2X	2X	3X	3X
PENDULUM HARDNESS, %	30.8	40.1	30.2	39.3
GEL FRACTION, %	93.6	94.6	93.4	94.5
CHEMICAL TEST (Yes/No)*				
Acetic acid, 5%	Y	Y	Y	Y
Ethanol, 50%	Y	Y	Y	Y
Sodium Carbonate, 1%	Y	Y	Y	Y
Sulphuric Acid, 10%	Y	Y	Y	Y
Sodium Hydroxide, 10%	Y	Y	Y	Y

\* Yes means the spot is not affected and vise versa.

Table 6: Natural rubber-based formulation for rubberwood substrates [27].

Liquid epoxidised natural rubber acrylate (LENRA-50)	20%
TPGDA	65%
TMPTA	10%
Benzophenone	2.5%
Irgacure 184	2.5%
Total	100%

Table 7: The properties and performance of the coating film from natural rubber-based resin (LENRA-50) [27].

Type of Test	Properties/performance
Viscosities (cps)	430
Gel content (%)	93.7
Gloss (%)	80.3
Pencil hardness	H
Pendulum hardness (%)	34.7
Adhesion (%)	82
Impact resistance [*]	3.2
Scratch resistance (kg)	1.44
Abrasion resistance	0.0036
Heat resistance [*]	5
Chemical resistance:	
Acetic acid 5%	good
Citric acid	good
Sulphuric acid 10%	good
Sodium carbonate 1%	good
Sodium chloride 10%	good
Sodium hydroxide 1%	good
Ethanol	good
Acetone	good

#9

RECOMMENDATIONS

&

#10

TERMINOLOGIES

**Recommendations**

During the seminar and workshop, some recommendations were made which were as follows:

**From participants:**

- i. list of the relevant suppliers of the resins and related chemicals should be made available to the participants.
- ii. more participants from wood/furniture manufacturing industry should participate in this kind of seminar and workshop.
- iii. workshop should also include curing technique for three dimensional substrates such as golf club heads, fishing rods and door frames.
- iv. include practical demonstration on the utilisation of a reverse roller coater.
- v. seek cooperations from universities for presentation of papers.
- vi. more time should be devoted to hands-on experience
- vii. include comparison studies on the finishing technologies with Malaysian perspectives.
- viii. give participants opportunities to coat their own products during practical demonstration.
- ix. due to its up-front capital cost and production volume, the emphasis should be given to the UV curing technology.
- x. encourage the manufacturing sector to use the UV curing technology
- xi. include in the itinerary a visit to a factory which use radiation curing technology.
- xii. conduct a seminar or workshop or even a course solely on UV curing technology

**From lecturers:**

- i. considering different levels of the background of the participants, separate them into several groups.
- ii. more participants from manufacturing sector should participate in the workshop.
- iii. organise the workshop for two groups of participants with concurrent sessions.
- iv. limit the number of participants to 15 persons in one group.

NATIONAL EXECUTIVE MANAGEMENT SEMINAR AND WORKSHOP ON SURFACE  
FINISHING BY RADIATION CURING TECHNOLOGY  
Kuala Lumpur and Bangi  
(7th - 9th., SEPT. 1993)

"RADIATION CURING FOR BETTER FINISHING"

Terminologies

Abrasion resistance : The ability of a material to withstand mechanical action such as rubbing, scraping or erosion, that tends to progressively remove material from the surface.

Absorbed dose : The absorbed dose (sometimes referred to simply as dose), D is the amount of energy absorbed per unit mass of irradiated matter. SI unit of absorbed dose is gray (Gy).

Acid curing (Hardening) : Process of curing or hardening resins through the use of acid catalysts. These are frequently employed with urea and melamine-formaldehyde resins.

Acrylics : Resins resulting from the polymerization of derivatives of acrylic acids, including esters of acrylic acid, methacrylic acid, acrylonitrile, and their copolymers. Also known as Acrylic Resins, and Acrylate Resins.

Actinic radiation : Electromagnetic radiation capable of producing photochemical action, but of insufficient energy to produce ions in a medium of common materials. Usually having wavelength of greater than 185  $\mu\text{m}$ .

Additive : Any substance added in small quantities to coating mixture, to improve properties.

Alkyds : Synthetic resins of great versatility, used as the basis of high quality paints, enamels and stoving finishes. Formed by the condensation of polyhydric alcohols with polybasic acids.

Ampere : Unit of electrical intensity (see Intensity).

Base : (a) A term used to indicate the major or characteristic ingredient in a particular paint, lead base, oil base, etc.  
(b) used to denote the ground or substrate which is to be painted/coated.

Binder : (a) Nonvolatile portion of the liquid vehicle of a coating. It binds or cements the pigment particles together and the paint film as a whole to the material to which it is applied. (b) Component of an adhesive composition which is primarily responsible for the adhesive forces which hold two bodies together. (c) Resin or cementing constituent of a

plastic compound which holds the other components together.  
(d) The components in an ink film which hold the pigment to the printed surface.

Cross-linking : The setting up of chemical links between the molecular chain of polymer.

Cure : Changing the properties of a polymeric system into a final, more stable, usable condition by the use of heat, radiation, or reaction with chemical additives.

Curing agent : Additive which promotes the curing of a coating.

Curing speed : It is indicated by the speed of conveyer belt on which the sample to be cured is carried.

$D_{min}$ ,  $D_{max}$  : Mean minimum and maximum absorbed doses in the product.

Dose meter : A device, instrument or system having a reproducible and measurable response to radiation that can be used to measure or evaluate the quantity termed absorbed dose, exposure or similar radiation quality.

Dose rate : Radiation dose per unit time.

Dose uniformity : The ratio of maximum to minimum absorbed dose in the product.

Dosimetry : The measurement of radiation quantities.

Double bond : A type of chemical bond wherein two pairs of electrons are shared between two atoms.

Electron : An electron is a negatively charged particle with a mass of  $9.21 \times 10^{-28}$  grams.

Electron beam machine : A device for producing a large amount of electrons with high kinetic energy.

Electron beam : An essentially monodirectional stream of (negative) electrons which have usually been accelerated electrically or electromagnetically to high energy.

Electron curtain : An electron beam generated via a linear source (or cathode), as opposed to a scanned source.

Electronbeam curing : Conversion of a liquid coating to a hard coating by means of electronbeam energy.

Electronvolt (eV) : A unit of energy. One electronvolt is the kinetic energy acquired by an electron in passing through a potential difference of one volt in vacuum.

**Emulsion** : A suspension of very small droplets of an oil, resin, bitumin or other liquid in water, usually brought about or stabilised by the aid of a third component called an emulsifying agent.

**Emulsion paint** : A paint in which the vehicle is emulsified in water. The term is usually applied to paints in which the dispersed binder is a synthetic polymer, most frequently polyvinyl acetate.

**Enamel** : Topcoat which is characterized by its ability to form a smooth surface.

**Epoxy resin** : A synthetic resin containing a chemical grouping (the epoxy group) conferring the ability to form coatings by various curing (hardening/drying) mechanisms.

**Extender** : A pigment of little opacity and colour used in paints to reduce cost or adjust the working or film forming properties such as film thickness, brushing and settling on storage.

**Filler** : A liquid or paste composition suitable for filling pores, cracks, joints and blemishes in surfaces, applied by brush, knife or coater.

**Flash point** : The temperature in degrees ( $^{\circ}\text{C}$ ) at which a liquid gives off sufficient vapour to form a flammable or ignitable mixture with air.

**Free radical** : Atom or molecule with an unpaired electron in the outer orbit.

**Functionality** : Effective number of bonds and points of intermolecular attachment in a chemical compound, the degree or order of reactivity.

**Gray (Gy)** : The SI unit of absorbed dose of ionising radiation, being to one joule of energy absorbed per kilogram of matter undergoing irradiation.

$$1 \text{ Gy} = 1 \text{ J/kg} = 100 \text{ rad.}$$

**Intensity** : The number of electrons per second available at the product (target) determines the intensity of the beam. The unit is the ampere (A), with the milli (mA) being the working level of a typical beam.

**Ionisation** : Production of ion pairs, one of which may be an electron.

**Ionising radiation** : Any radiation, consisting of directly or indirectly ionising particles, or a mixture of both.

Line speed : see curing speed.

Mar resistance : The ability of the surface of a material to withstand mechanical forces.

Mer : The smallest repeating structural unit of any high polymer.

Mercury lamp : Lamp in which light is generated through presence of mercury vapour. Most UV lamps are mercury vapor lamps.

Monomer : Substance composed of low molecular weight molecules capable of reacting with like or unlike molecules to form a polymer.

MSDS (Material Safety Data Sheet) : A document, available from suppliers of chemical products, which outlines potential hazards associated with the particular chemical product and methods for proper handling.

Oleo-resinous : A term applied to media composed of oils and natural (or modified natural) resin, usually in contrast to alkyd and synthetic resin media.

Oligomer : A polymer composed of molecules containing only two, three or a few mers

Orange peel: Surface conditions characterized by an irregular waviness of the coating material, resembling an orange skin texture.

Overcure : Caused by an aftercure or by being subjected to too high a temperature, too long a period at proper temperature or too long exposure to irradiation and resulting in a product less resistant to aging.

Paint : A suspension of finely divided solid pigment in an oil or varnish which is applied to a surface as a liquid and becomes converted to a solid film.

Photocurable Coating : A polymerizable mixture that can be applied as a thin film to a substrate and polymerized at rapid rate by exposure to actinic light..

Photoinitiator : A substance which absorbs radiant energy, and undergoes a chemical process, which results in reactive intermediates capable of beginning the polymerization process.

Polymer : A complex molecule formed by the chemical linkage of a large number of individual units (monomers).

Polymerization : Chemical reaction in which two or more small molecules (monomers) combine to form large molecules (polymers) that contain repeating structural units of the



original molecules.

Post cure : Continuation of reactions of materials in the ink or coating after exposure to radiation has ceased.

Primer : The first coat of a paint or coating system used on a specific surface to provide adhesion, satisfy absorption or prevent corrosion.

Radiation : The propagation of energy through space.

Rad (rad) : The special unit of absorbed dose. The rad is defined as :

$$1 \text{ rad} = 0.01 \text{ Gy} = 0.01 \text{ J/kg} (= 100 \text{ erg/gm})$$

Radiation source : An apparatus or radioactive substance in a suitable support that constitutes the origin of the ionizing radiation (e.g. cobalt-60 source rods in a frame, or an electron beam machine or accelerator).

Reactive diluent : A viscosity reducer for coating which has low volatility and will become permanent part of the coating through chemical reaction.

Resin : The term normally used to designate any polymer that is a basic material for coatings and plastics.

Sealer : A clear or pigmented composition used to prevent excessive suction of porous surfaces or to prevent soluble matter or alkali in the surface from bleeding into or affecting subsequent coats.

Shelf life : The amount of time a material may be stored under specified conditions with no significant changes in properties.

Solids content : Percentage by weight of nonvolatile matter in a coating, adhesive or other aqueous dispersion.

Solvent : A volatile liquid used to dissolve the binder or resinous component, and lost from the film by evaporation during drying.

Substrate : The material to be coated.

Synergism : Property by which two or more agents act together more effectively than either alone.

Thinner : A volatile solvent used to reduce the viscosity of paints or paint vehicles to the correct degree for application. Water is the thinner in emulsion paints.

Thixotropy : The property of reversible change in some materials

from solid to liquid induced by mechanical agitation.

Topcoat : The coating intended to be the last coat applied in a coating system; usually applied over a primer, undercoaters or surfacers. Also known as finish coat.

Undercoat : The coat(s) intermediate between primer (or existing paint) and finishing coat. Not to be confused with "primer".

Undercure : The opposite of overcure. May be evidenced by tackiness, longness, or inferior physical properties.

Ultraviolet curing : Conversion of liquid coating to a hard coating by means of ultraviolet light.

Varnish : An oleo-resinous or synthetic resin medium without pigment used to improve the appearance or durability of a surface or to increase the gloss of an oil paint system.

Vehicle : The liquid portion of a paint in which the solid pigment is dispersed.

Window : A metallic foil in an electron beam generating unit that allows passage of energetic electrons from the beam.

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