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Comparison of Physical Properties of Coatings on Paper Substrate after Curing by UV and Electron Beam

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Abstract

This study aimed to assess the curing efficacy of ultraviolet (UV) radiation and electron beam (EB) curing techniques and the physical attributes of coated overprint varnish (OPV) applied on paper substrates. Coating formulations with and without photo-initiator (PI) underwent UV irradiation at doses ranging from 0.561 to 1.612 J/cm^2 and EB at doses ranging from 30 to 100 J/g. The investigation utilized Fourier-transform infrared (FTIR) spectroscopy to analyze the degree of conversion of double bonds in the cured samples, with a notable absorption peak observed at 810 cm^{-1} corresponding to C=C bonds in the acrylate group. Furthermore, EB curing exhibited polymer curing percentages ranging from 79% to 83%, similar to UV curing. Notably, the yellowness index of EB-cured coating films (ranging from 0.5 to 10) was lower than that of those cured with UV (ranging from 10 to 28). The study suggests that EB curing could serve as an alternative process, potentially reducing health hazards associated with photo-initiator migration and food contamination within packaging materials, thereby offering a promising future for the field.

Keywords: Electron Beam, UV Curing, EB Curing, Yellowness, Coatings

1. Introduction

In the packaging industry, the adhesion, transparency, and color of the packaging are critical to product appearance. The UV overprint varnish (UV-OPV), which consists of oligomer, monomer, additive, and photo-initiator (PI), is applied to increase gloss for a better appearance and protective barrier from abrasion and tear due to handling or contact with moisture, chemical, or other potential sources of damage (1).

To achieve the coated layer, UV rays (2) and electron beams (EB) (3) are selected as the light sources for polymer curing, which includes ink curing and coated curing.

Compared with traditional thermal and UV curing, EB curing is green technology, environmentally friendly, energy-efficient, and reliable for coatings, paints, and composite materials because of its high conversion, good surface adhesion, processing efficiency, and high throughput (4-9).

Curing is a process that transforms a monomer in liquid form into a 3-dimensional network of polymer in solid form by the polymerization and cross-linking reaction.

Currently, the production rate in the industrial field of UV curing is higher than in the EB curing process (10-11). The crosslinking structure results from the interaction between vinylidene groups and alkyl radicals, according to the mechanism shown in Figure 1, reprinted from Abdelhafidi et al. (2015) (12)

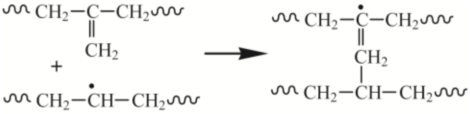

Figure 1 Crosslinking reaction between a vinylidene and an alkyl radical.

Currently, a study on the effect of the reactive group of urethanes di(meth)acrylates on the EB curing process has been proposed (8). It has been proven that acryloyloxy groups showed a higher polymerization rate of double bond conversion than methacryloyloxy groups.

However, UV-OPV has a high potential to form crosslinking networks with surfaces and increase their properties. However, the UV-cured sample shows a yellowing surface appearance. This phenomenon can be explained by the fact that PI may cause yellowing during long aging periods and under photolytic aging conditions (e.g., UV rays and fluorescent light) (13). In addition, the appearance of yellowing is affected by several other factors, such as temperature, moisture, and oxidation reaction (14-15). The yellowing discoloration is undesirable and has become anathema in the industry. Hence, PI resulting in yellowing would not sufficiently meet the most stringent industry demands (16-17).

To avoid the yellowing effect, the curing process of EB is an outstanding process that provides ionizing radiation. PI is not necessary in this system because the EB, which has higher energy than UV rays, can produce free radicals to form a three-dimensional network of polymer. In the last decade, the cost of the EB process has significantly reduced, which provided a slightly well-known increase in EB usage in some printing and packaging industries (6, 18) reported that 70%v/v epoxy acrylate oligomer was combined with 20% v/v 1,6-hexanediol diacrylate monomer and 10% v/v castor oil to produce the coating solution. The mixture solution was coated on the printed substrate and cured with various doses of EB (1, 2.5, and 5 Mrad pass^{-1}). After passing through the EB curing process, the coated layer was dried and formed like thin films. The thin film layer showed good adhesion between the coated layer and the printed substrate and no significant difference when compared with the UV curing process.

In 2013, Shin et al. studied the crosslinking structure between 75% v/v bisphenol-A-type acrylate epoxy oligomer and 25% v/v tripropylene glycol diacrylate on with and without substrate under various doses of EB radiation (10 MeV, 0-600 kGy). The result found that the cured layer showed a good curable degree at 40 kGy of EB dose. (19)

In 2020, Liu et al. polymerized the urethane di(meth) acrylate with different terminal groups (i.e. acryloyloxy and/or methacryloyloxy group) by EB curing. The result found that both terminal group resins could be crosslinked under EB irradiation without PI. (8)

According to the advantages of the EB curing process, this study aims to clarify and compare the properties of the coated layer between UV and EB curing processes on OPV coating. The physical properties of yellowness and whiteness, chemical properties of functional group and double bond conversion, and morphology of OPV coated sample were intensively characterized. This research presents the use of EB technology in drying varnish coatings in the packaging printing industry instead of using traditional UV technology that is unsafe. Because they use chemicals that are harmful to humans. Currently, EB technology is not widely used in the packaging printing industry.

2. Materials and Experiment

2.1 Materials

A commercial grade of Overprint varnish (OPV), including Epoxy acrylate (EA), tripropylene glycol diacrylate (TPGDA), and Benzophenone (BP), was purchased from Anwill Co. Ltd. (Bangkok, Thailand). The chemical structures of TPGDA, BP, and EA are illustrated in Figure 2. This research selected tripropylene-glycol diacrylate (TPGDA), epoxy acrylate (EA), and benzophenone (BP) because they are the primary components commonly used by OPV manufacturers. All chemical substances, such as chloroform, dichloromethane, and methanol, were analytical grade purchased from Lab Scan, Co., Ltd. (Bangkok, Thailand).

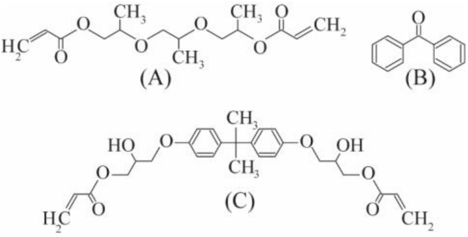

Figure 2 Chemical structures of (A) tripropylene glycol diacrylate; TPGDA, (B) Benzophenone; BP and (C) Epoxy acrylate; EA.

2.2 Preparation of OPV coating solutions

Approximately 20 ml of OPV solution was prepared in 7 formulations, which varied the TPGDA monomer (57-59 %wt), EA oligomer (37-39 %wt), and BP photo-initiator (0-6 %wt) ratios as well as varying the curing process between UV and EB. The name, composition, and curing process of 7 formulations are presented in Table 1. Each OPV solution was mixed and stirred continuously in an ambient atmosphere until the solution was homogeneous. Consequently, the solutions were coated on paper with a wet film thickness of 40 µm using k-bar coater No. 4.

Table 1 OPV formulations for coating paper

Sample name	OPV components			Curing process
	TPGDA (wt%)	EA (wt%)	BP (wt%)	
UV-OPV-A	59	39	2	UV
UV-OPV-B	58	38	4	UV
UV-OPV-C	57	37	6	UV
EB-OPV-A	59	39	2	EB
EB-OPV-B	58	38	4	EB
EB-OPV-C	57	37	6	EB
EB-OPV-NO	60	40	None	EB

2.3 Preparation of OPV coated paper using UV and EB irradiation process

After coating various OPV solutions, each coated sample was irradiated using UV and EB to form the coated layer by the curing process. For UV irradiation, the coated samples (UV-OPV-A, UV-OPV-B, and UV-OPV-C) were cured under UV from a medium-pressure mercury vapor lamp source in atmospheric conditions. The UV irradiated doses were selected at 0.561, 0.672, 0.797, 1.000 and 1.612 J/cm^2. The UV energy is measured by a UV dose meter, RUNWING UV-MINI 250-410 nm. For EB irradiation, the coated samples (EB-OPV-A, EB-OPV-B, EB-OPV-C, and EB-OPV-NO) were cured using an Electron beam (NHV corporation, Japan) at 150 keV in the nitrogen atmosphere. The EB irradiated doses were selected at 30, 50, 70, and 100 kGy.

2.4 Chemical properties

The functional group of coated paper with various OPV formulations was determined using Fourier transform infrared spectroscopy (FTIR) (Bruker Tensor 27; Billerica, MA). Before testing, the coated samples were dried in a hot air oven at 60 ^{C} for 24 h. All samples were tested in attenuated total reflection mode (ATR mode). The FTIR spectra were recorded at wavenumber 4000-650 cm^{-1} at 32 scanning numbers and 2 cm^{-1} of resolution. In this characterization, the comparison of crosslinking efficiency between UV and EB curing processes was also determined using the degree of double bond conversion (DC) (8, 20). The percentage of DC was calculated as in equation 2.1,

$$DC = 1-[\{A_{(C=C)}/A_{(C=O)}\}_t/\{A_{(C=C)}/A_{(C=O)}\}_0] \times 100 \qquad (2.1)$$

where $A_{(C=C)}$ is the integration area of the acrylate group (C=C) of samples before and/or after irradiation at the different doses, and $A_{(C=O)}$ is the integration area of a carboxyl group (C=O) of samples before and/or after irradiation at the different dose, t is the integrated area after irradiation with different doses, 0 is the integrated area before irradiation.

2.5 Morphology property

The cross-section morphology of coated paper with various OPV formulations was observed using a scanning electron microscope (SEM) (JEOL JSM-5410LV; Tokyo, Japan). All coated samples were frozen with liquid nitrogen and fabricated into approximately 1.00 x 1.00 cm square shapes. Before observation, the shaped sample was coated with gold to prevent charging from the specimen's static electric fields. All samples were observed at 15 kV of acceleration voltage and 6,000X magnification.

2.6 Color analysis

The color changing of the yellowness index (YI) of coated paper with various OPV formulations was investigated using an X-Rite Color i5 spectrophotometer (X-Rite, USA). All samples were tested in the CIELAB system (D65/10°, specular component included) according to ASTM E313. The color value was observed at three random locations of each sample and calculated to YI using equation 2.2.

$$YI = (1.301\,X_{10} - 1.149 \cdot Z_{10})/Y_{10} \qquad (2.2)$$

where X_{10}, Y_{10}, and Z_{10} are the tristimulus values at 10° standard observer.

3. Results and Discussion

Seven formulations of coating solutions in this work contained 57-59 wt% tripropylene-glycol diacrylate (TPGDA) as a monomer, 37-39 wt% epoxy acrylate (EA) as an oligomer, and 0-6 wt% benzophenone (BP) as a photo-initiator. After irradiation with UV and EB, the OPV solution formed a 3-dimensional network polymer. This polymer appeared as a solid thin layer on the paper substrate due to the polymerization and crosslinking reactions. Figures 3 and 4 present the physical appearance of paper coated by OPV at various conditions. The UV curing process in Figure 3 displays the color appearance with more yellowing than the EB curing process, as shown in Figure 4. Moreover, the more UV dose and PI concentration additions, the more yellowing appearance in the coated layer.

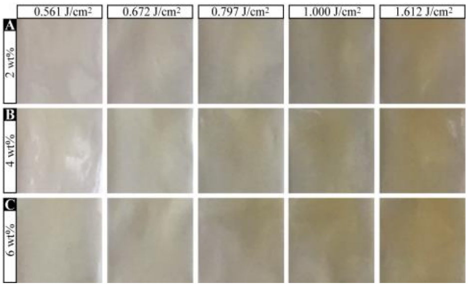

Figure 3 Physical appearance of UV-OPV-A (top), UV-OPV-B (middle), and UV-OPV-C (bottom), which are curing at various UV doses (0.561 J/cm^2, 0.672 J/cm^2, 0.797 J/cm^2, 1.000 J/cm^2, and 1.612 J/cm^2).

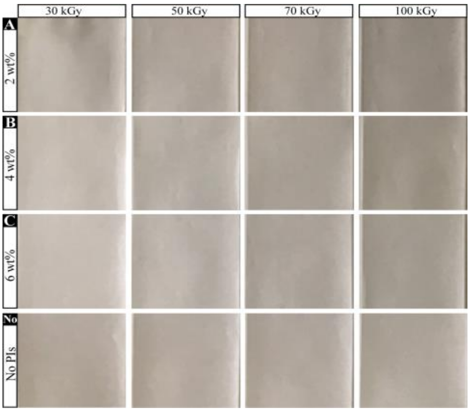

Figure 4 Physical appearance of EB-OPV-A (EP 2 wt%), EB-OPV-B (EP 4 wt%), EB-OPV-C (EP 6 wt%), and EB-OPV-NO (no BP), which are cured at various EB doses of 30 kGy, 50 kGy, 70 kGy, and 100 kGy.

3.1 Chemical properties

FTIR was used to characterize the chemical group of coated samples. Figure 5 shows the overall spectra of uncured OPV, UV-OPV-A, EB-OPV-A, and EB-OPV-NO. The FTIR spectra of OPV-coated samples cured with UV and EB are demonstrated in Figures 6 and 7, respectively. The results showed that the vibration of C=O in the carboxylic group and C–O ester stretching, which appeared at 1730 cm^{-1}, was found in all samples. The peaks at 1197 cm^{-1} and 1724 cm^{-1} correspond to the stretching vibration of the C–O–C of the ester groups and the C=O of the carbonyl groups, respectively. The 984 cm^{-1} peak corresponds to the stretching vibration of the C=C-C=O. Other bands included the C–H stretching vibration of alkane at 1453, 2981-2879 cm^{-1}. The vibration of C=C in double bonds from various peaks at 1635 and 1409 cm^{-1} was indicated by the existence of an acrylate group from TPGDA and EA, which is mostly found in uncured OPV. The intensity of UV irradiation has a direct impact on the FTIR curve by altering the chemical structure and composition of the material being studied. This can be reflected in the changes in peak intensities and positions and the emergence of new peaks. In our study, they hardly change with the UV intensities. Additionally, the peak around 810 cm^{-1} observed in only the uncured sample was represented by C=C stretching in the acrylate group of TPGDA and EA. The peak at 810 cm^{-1} of uncured OPV spectra decreased after UV and EB irradiation. This phenomenon could be explained for two reasons based on the light source. The first reason is that the UV and EB could activate the PI in UV-OPV-A and EB-OPV-A, which consequently debonded PI and followed the PI radicals. Afterward, the radicals could promote the propagation step of chain polymerization by monomer and oligomer combination. Another reason is that the EB could generate acrylate radicals from the acrylate group in uncured OPV. These radicals could also promote the propagation step of chain polymerization, as mentioned in the first reason. To conclude, the disappearance of the 810 cm^{-1} peak, representing the C=C stretching of the acrylate group, after E-beam or UV irradiation indicates that the double bonds have broken and formed free radicals. These radicals react with other monomers or oligomers to form new C-C bonds, creating a three-dimensional crosslinked polymer network.

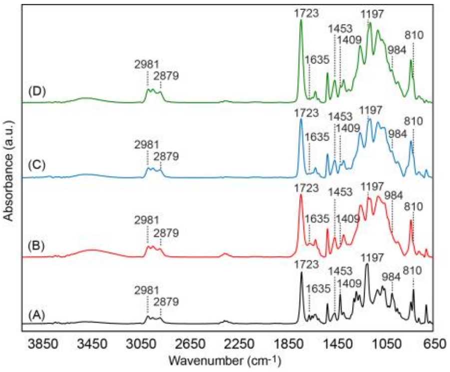

Figure 5 FTIR spectra of uncured (A) OPV, (B) UV-OPV-A, (C) EB-OPV-A, and (D) EB-OPV-NO.

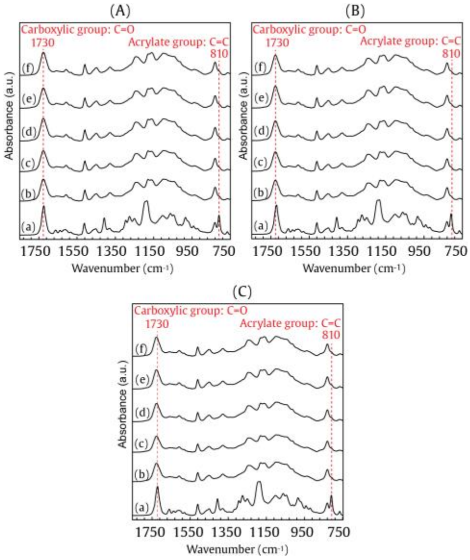

Figure 6 FTIR spectrum of (A) UV-OPV-A, (B) UV-OPV-B, and (C) UV-OPV-C at various UV doses (a) unirradiation, (b) 0.561 J/cm^2, (c) 0.672 J/cm^2, (d) 0.797 J/cm^2, (e) 1.000 J/cm^2 and (f) 1.612 J/cm^2.

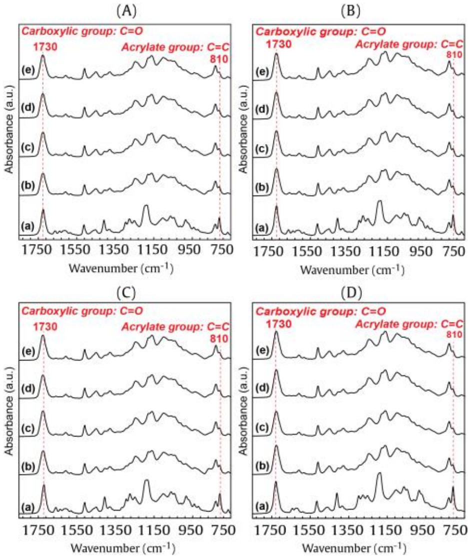

Figure 7 FTIR spectrum of (A) EB-OPV-A, (B) EB-OPV-B, (C) EB-OPV-C, and (D) EB-OPV-NO at various EB doses of (a) un-irradiation, (b) 30 kGy, (c) 50 kGy, (d) 70 kGy and (e) 100 kGy.

Figure 8 presents the percentage of DC in various PI concentrations under UV and EB curing processes. The DC was used to identify the performance of the curing process between UV and EB curing. For the UV curing process, the results showed that the irradiated OPV-coated paper exhibited high crosslink density after irradiation with the UV dose in the range of 0.561-1.612 J/cm^2. In a range of 0.561-1.612 J/cm^2, the coated paper showed approximately 76% DC with no significant difference between various UV doses. For the EB curing process, the results showed that the irradiated OPV-coated paper exhibited high crosslink density after irradiation with the EB dose in the 30-100 kGy range. In a range of 30-100 kGy, the coated paper showed approximately 76-85% of DC depended on PI content, as mentioned later.

For UV radiation, the organic materials can degrade when exposed to UV light. The degradation can lead to the breaking of chemical bonds, resulting in the formation of new functional groups. Considering the polymer or resin materials, the UV irradiation can break polymer chains, leading to the formation of carbonyl (C=O) groups, which show up as new peaks in the FTIR spectrum around 1700 cm^{-1} or the C-H stretching peak might decrease due to the breaking of C-H bonds.

A comparison between various concentrations of PI (0, 2, 4, and 6 wt%) from 7 OPV formulations was studied. For UV curing, the additional PI at various contents presented 76% of DC with no significant difference at various concentrations. On the other hand, the EB-OPV showed more DC performance than UV-OPV. The percentage of DC in OPV samples at various PI after passing through the EB curing process presents the increase of DC (76-85%) when increased PI (0-6 wt%). The double radical initiation from PI substance and EB generation could explain this phenomenon. Moreover, the non-oxidative atmosphere promoted a high level of cross-linking between the molecules in the formulation because the oxidation reaction in a normal atmosphere could obstruct the DC performance (21). When comparing the performance of DC between UV and EB, the results exhibited that the degree of DC in EB-OPV-NO was similar to UV-OPV at various PI. Thus, replacing UV curing in OPV with PI by EB curing in OPV without PI was a good practice to reduce the PI migration from printed food contact materials to food and the contamination of PI in the environment.

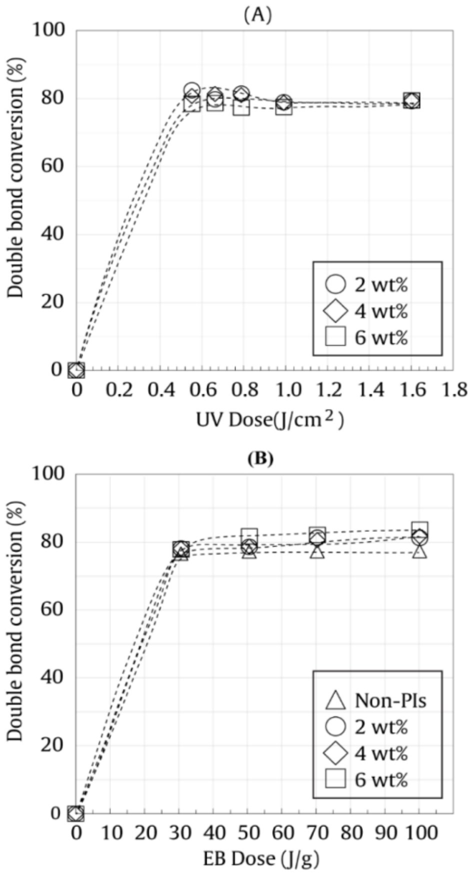

Figure 8 Double bond conversion (%) of OPV coated paper with various (A) UV doses, (B) EB doses, and different PIs concentrations of 2 wt% (○) 4 wt% (□), and 6 wt% (◇) and non-PIs (△).

3.2 Morphology property

The cross-section morphology of uncoated and coated paper with OPV at various light sources was used to determine the compatibility between the coated layer and paper substrate, as illustrated in Figure 9. The results showed that EB-OPV-A coated paper (Figure 9C) displayed good surface adhesion, the same as that of UV-OPV-A coated paper (Figure 9B). It can be seen that the UV radiation's coating on the paper showed some thin film disjoined on the paper.

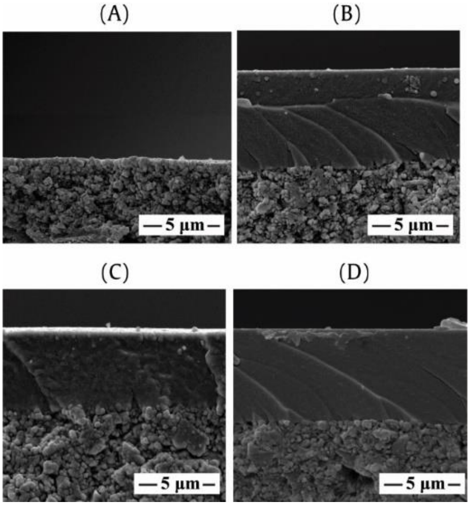

Figure 9 SEM images of (A) uncoated paper, (B) coated paper with UV-OPV-A, (C) with EB-OPV-A, and (D) with EB-OPV-NO.

The EB-OPV-coated paper without PIs showed smooth and excellent adhesion after irradiation at 30 J/g. Compared with different PI concentrations, the morphology in the cross-section of UV-OPV coated paper at 4 and 6 wt% PIs displayed better and smoother adhesion than 2 wt% in Figure 10A(a-c). Meanwhile, the EB-OPV coated paper showed excellent adhesion and smoothness in only 2 wt% of PIs (Figure 10B(a-c)).

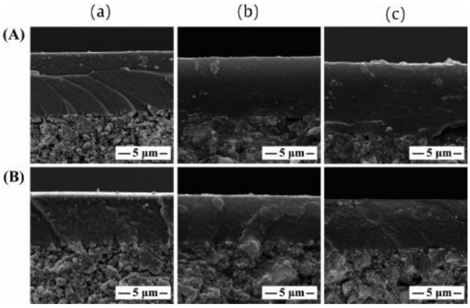

Figure 10 SEM image of (A) UV irradiation and (B) EB irradiation with the different PIs concentrations (a) 2 wt%, (b) 4 wt%, and (c) 6 wt%.

3.3 Color analysis

The color changing of coated paper with various OPV formulations was determined in terms of yellowness and whiteness indexes, as shown in Figures 11 and 12. Figure 11(A-B) shows the yellowness index of UV-OPV and EB-OPV. The yellowness index of the initial sample was 0.716. After irradiation, the surface appearance displayed an increase in the yellowness index. In addition, the more UV dose irradiation, the more the yellow index increases. This finding was agreed with Arsu et al. (1995) (21) and Studer et al. (2001) (22). The yellowing phenomenon could be explained by the oxidation reaction of the crosslinked polymer under a normal atmosphere in UV irradiation. The oxidation reaction might break the covalent bonding of epoxy resin and promote the chromophore group, such as carbonyl groups, double bonds, and conjugated structures, which provide a yellow color. On the contrary, EB curing, processed in the nitrogen atmosphere, showed a lower yellowing index (lower than 11 based on PI concentration) of EB-OPV compared with UV-OPV. This was because the non-oxidative atmosphere might promote less chromophore group (22). The PI concentration increase in UV-OPV and EB-OPV affected the yellowness index. On the other hand, EB-OVP-NO displayed the lowest yellowness index, which was less than three after EB irradiation.

The whiteness index result showed an inversely variable YI value. The whiteness index of UV-OPV and EB-OPV was lower than that of the uncoated sample (approximately 115). For UV-OPV, the whiteness index was lower (76 to 31) when the UV dose was higher (0.561 J/cm^2 to 1.612 J/cm^2), as shown in Figure 12A. The EB affected the whiteness index less than UV, which presented a whiteness index in the range of 70-100, as shown in Figure 12B.

Electron beam (EB) irradiation has a lesser impact on the whiteness index of OPVs than UV irradiation due to distinct variations in the mechanisms and effects of these two curing processes. EB curing does not require PIs, unlike UV curing, which relies on PIs to initiate the polymerization process.

Over time, PIs in UV curing can undergo yellowing or discoloration, diminishing the whiteness index of the OPV. The exclusion of these substances in EB curing mitigates this outcome. EB irradiation directly transfers energy to the material, inducing ionization and excitation of molecules that result in polymerization. This method is more regulated and does not entail the photodegradation of additives or substrates, thereby conserving the original color and whiteness of the material.

Unlike UV irradiation, which can lead to the yellowing of specific substrates and additives due to prolonged exposure to high-energy UV radiation, EB irradiation is less prone to causing such yellowing since it does not emit UV radiation and thus avoids this form of photochemical degradation. Normally, the UV lamps used to dry coatings are heated up to 100 degrees, which causes the coating layer to degrade.

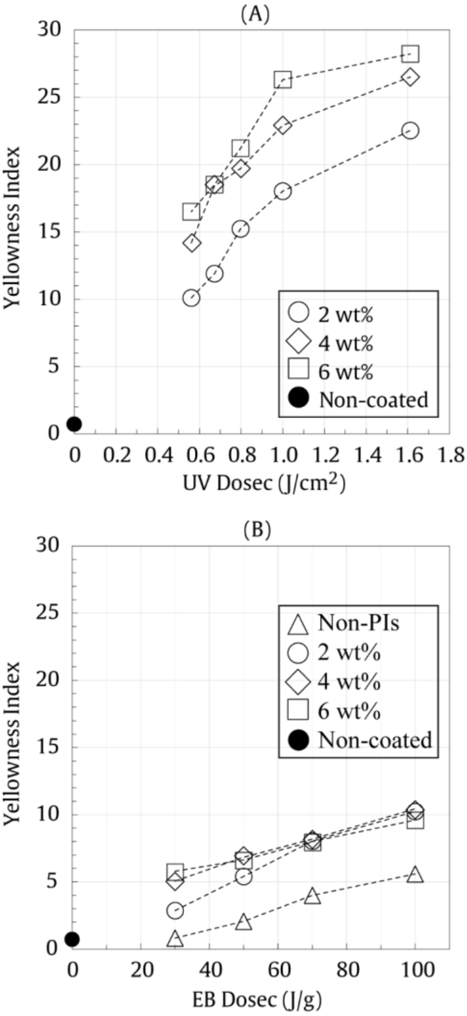

Figure 11 Yellowness index of OPV coated paper with various (A) UV doses, (B) EB doses, and different PIs concentrations of 2 wt% (○) 4 wt% (□), and 6 wt% (◇) and non-PIs (△).

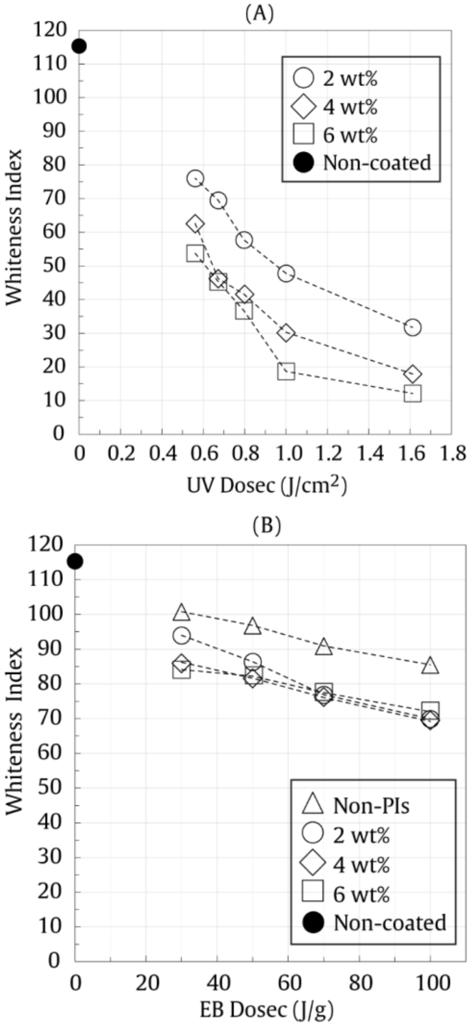

Figure 12 Whiteness index of OPV coated paper with various (A) UV doses, (B) EB doses, and different PIs concentrations of 2 wt% (○) 4 wt% (□), and 6 wt% (◇) and non-PIs (△).

In EB curing, precise management of the penetration depth of electrons is achievable, ensuring that the curing primarily occurs at the surface or intended layer. This targeted application of energy minimizes thermal and photochemical consequences that could result in discoloration.

Compared to UV curing, EB curing produces less heat, lowering the likelihood of thermal degradation of the varnish and the substrate. Excessive heat can induce discoloration, adversely affecting the whiteness index.

Assessing the efficacy of ultraviolet (UV) radiation and electron beam (EB) curing methods for overprint varnish (OPV) on paper substrates presents various challenges and opportunities for future investigation. One notable challenge pertains to optimizing curing parameters, such as printing speed and curing unit configurations, which are essential for achieving proper curing, strong adhesion, and minimal component migration, particularly in food packaging scenarios (23). Furthermore, although UV LEDs offer benefits such as reduced energy consumption and the absence of ozone production, they may not be suitable for formulations intended for mercury lamps, highlighting the necessity for additional research on UV LED-compatible varnishes (25). Conversely, EB curing exhibits potential in achieving thorough curing at specific levels and the possibility of decreasing synthetic chemicals through the integration of vegetable oils; however, the high initial expenses and complexity associated with EB equipment act as impediments (24). Additionally, the migration of photoinitiator molecules in UV-cured coatings presents obstacles for food packaging compliance, indicating that more effective post-irradiation techniques like DirectCure could be investigated to mitigate migration concerns (26). The physical properties of cured OPV, including adhesion, flexibility, and resistance to various chemicals, play a crucial role in the longevity and safeguarding of printed materials. However, current research predominantly concentrates on specific characteristics like gloss and surface adhesion, underscoring the need for comprehensive examinations of long-term stability and environmental impacts (27). Prospective studies may also explore the creation of hybrid curing systems amalgamating the advantages of both UV and EB technologies alongside the investigation of novel bio-based materials to enhance OPV sustainability. Despite significant advancements, there remains extensive potential for furthering the comprehension and utilization of these curing techniques to meet the evolving standards of the industry and environmental concerns.

EB curing has broad impacts, such as environmental and health benefits, including reduced emissions and energy efficiency. It also prevents ozone formation and improves workplace safety. Additionally, it offers material versatility and faster processing times, leading to increased production efficiency. Industries can benefit from these advantages by adopting EB curing.

4. Conclusions

The study lies in its comprehensive comparative analysis, methodological rigor, and emphasis on the practical and safety benefits of EB curing over UV curing, particularly in the context of overprint varnish applications on paper substrates. We compared the properties of paper coated with overprint varnish using UV and EB curing processes. Analysis of FTIR spectra revealed the disappearance of the C=C stretching peak in the acrylate group of TPGDA and EA around 810 cm-1 in cured samples, indicating successful polymerization and crosslinking of the monomer and oligomer. The degree of double bond conversion in the EB curing process (76-85%) exceeded that of UV curing (76%) with higher levels of PI, contributing to higher conversion rates.

Furthermore, the crosslinked layer of overprint varnish cured by both UV and EB methods exhibited excellent compatibility with the paper substrate, with no significant differences observed. However, the yellow index of EB-cured samples (ranging from 0.5 to 10) was lower than that of UV-cured samples (ranging from 10 to 28), and the addition of PI intensified the yellow coloration. Conversely, the whiteness index demonstrated an inverse relationship with the yellow index.

Considering these findings, EB radiation emerges as a promising alternative to UV curing in surface coating applications. It offers the potential to mitigate the risks of PI migration and environmental contamination associated with UV curing. This emphasizes the viability of EB curing as a safer and more efficient method for the surface coating industry, particularly in contexts where food safety and environmental concerns are highest.

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Declaration of Conflicting Interests

The authors declared that they have no conflicts of interest in the research, authorship, and this article's publication.

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