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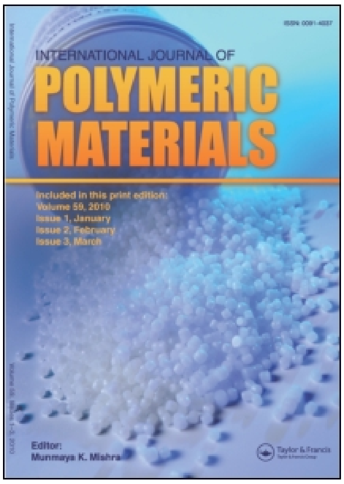

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The Effect of Radiation Dosages and UV/EB Radiation on the Properties of Nanocomposite Coatings

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The aim of this study was to compare the effects of ultraviolet (UV) and electron beam (EB) radiation on the properties of cured nanocomposite coatings. Surface hardness increased with increasing radiation dosages (number of passes) for all samples. This was due to the increase in crosslinking with increasing radiation dosages. Pendulum hardness, gel content, and thumb twist results were analyzed to choose the appropriate curing dosage for both curing techniques. The selected dosages were then used to cure coatings for scratch and abrasion resistance tests. It was found that the UV curing produced coatings with better abrasion resistance, whereas EB curing was more suitable for producing scratch-resistant coatings.

Keywords: EB radiation, nanocomposite coatings, radiation dosages, UV radiation

INTRODUCTION

Radiation curing refers to the hardening of a polymer material by crosslinking of polymer chains, brought about by radiation techniques such as ultraviolet radiation (UV) and electron beam (EB). The process involves transformation of a liquid substance into a non-tacky solid in less than 1 s or fraction of a second [1]. Radiation curing has grown within the last 30 years from being a novelty to a firmly established, credible alternative to many of the traditionally cured systems [2].

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This is largely due to improved productivity, increased product performance, and elimination of hazardous volatile organic compounds (VOC). Among all radiation curing techniques available today, UV and EB curings are the most widely practiced.

The EB-induced free radical polymerization of vinyl and acrylic monomers is considered to follow the same reaction scheme as for the UV-induced polymerization. The main difference between the two initiation processes lies in the energy deposition and the pathway of free radical generation [3]. EB-cured coatings are usually uniformly cured due to in-depth penetration of electrons. This renders the EB-cured coatings hard but brittle. Meanwhile, its UV counterparts tend to be less "cured" due to the photoinititiators which absorb the UV radiation at the surface, resulting in the surface having the highest radical concentration [2]. This will lead to the formation of a polymeric network with lower molecular weight. Consequently, this will affect the surface hardness of the coatings. Based on these differences, the effect of UV/EB radiation on the scratch and abrasion resistance of coatings could be established.

Many research works dedicated to nanocomposite coatings utilized either UV curing or to a lesser extent, EB curing techniques. Zahouily et al. [4], Tsukruk et al. [5], Decker et al. [6], and Amerio et al. [7] were among those who studied UV-cured coatings. On the other hand, Xuecheng et al. [8] and Kumar et al. [9] concentrated on EB-cured coatings. Only a few compared UV/EB coatings directly, as demonstrated by the works of Patacz et al. [10] and Ruiz et al. [11]. However, no study has yet been reported on the effect of UV/EB radiation on the scratch and abrasion resistance of nanocomposite coatings. The aim of the present work is to fill in the gap in this area.

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EXPERIMENTAL

Materials

The raw materials (Table 1) selected to produce radiation-curable material consist of prepolymer (oligomer), monomer (reactive diluent), coupling agent, filler, catalyst, stabilizer, and photoinitiators (for UV-cured coatings only). In this research, urethane acrylate prepolymer (Ebecryl 230) was used to impart flexibility while epoxy acrylate prepolymer (Ebecryl 600) was used to impart good hardness and abrasion resistance to the coatings. Tripropyleneglycol diacrylate (TPGDA) and pentaerythritol tri/tetraacrylate (PETIA) monomers were used to increase flexibility and crosslink density of cured film, respectively. The prepolymers and monomers used were purchased from UCB Chemicals, Belgium.

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TABLE 1 Formulations and Temperature for Synthesis

Materials	F1	F2	F3	F4	F5	F6	F7	F8	F9	F10	F11	F12	F13	F14
Ebecryl 230	45	35	45	35	17.5	–	–	–	–	–	–	–	–	–
Ebecryl 600	–	–	–	–	17.5	45	40	35	30	25	20	15	15	10
TPGDA	30	30	–	–	–	–	–	–	–	–	–	–	15	15
PETIA	–	–	30	30	30	30	30	30	30	30	30	30	15	15
VTMOS	25	25	25	25	25	25	25	25	25	25	25	25	25	25
Aerosil OX-50	–	10	–	10	10	–	5	10	15	20	25	30	30	35
	100	**100**	**100**	**100**	**100**	**100**	**100**	**100**	**100**	**100**	**100**	**100**	**100**	**100**
Water	–	25 g	–	25 g	25 g	–	25 g	25 g	25 g	25 g	25 g	25 g	25 g	25 g
Maleic anhydride	–	1.5 g	–	1.5 g	1.5 g	–	1.5 g	1.5 g	1.5 g	1.5 g	1.5 g	1.5 g	1.5 g	1.5 g
4-Hydroxy anisole	480 mg	480 mg	480 mg	480 mg	480 mg	480 mg	480 mg	480 mg	480 mg	480 mg	480 mg	480 mg	720 mg	720 mg
Irgacure 500	2%	2%	2%	2%	2%	2%	2%	2%	2%	2%	2%	2%	2%	2%
Darocur 1173	1.5%	1.5%	1.5%	1.5%	1.5%	1.5%	1.5%	1.5%	1.5%	1.5%	1.5%	1.5%	1.5%	1.5%
Temperature for synthesis	65°C	65°C	65°C	65°C	65°C	65°C	65°C	65°C	65°C	65°C	65°C	65°C	60°C	60°C

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Vinyltrimethoxysilane (VTMOS) coupling agent, often used to enhance the interaction between silica nanoparticles and acrylate matrix, was selected in this study. According to Salleh et al. [12], nanocomposites treated with VTMOS showed higher abrasion resistance than propyltrimethoxysilane (PTMOS). Silica nanofiller (Aerosil OX-50) was used as reinforcing filler. This filler has low thickening and agglomeration properties compared to other types of Aerosil available in the market. The VTMOS and Aerosil OX-50 used were purchased from Degussa-Hüls, Germany.

Maleic anhydride was used as a catalyst in this study to induce crosslinking and compatibility [13]. The amount of maleic anhydride and water used for dilution process was based on previous work [12]. The diluted maleic anhydride was added during the synthesis of radiation-curable materials. 4-hydroxy anisole stabilizer was also added during the synthesis to prevent premature polymerization caused by the heat and shearing action of the rotating blades. The amount used was also based on previous work [13]. Both maleic anhydride and 4-hydroxy anisole were purchased from Sigma-Aldrich, UK.

For UV-cured coatings, the inclusion of photoinitiators into the formulations was necessary. Irgacure 500 and Darocur 1173 photoinitiators purchased from the CIBA Switzerland were used for UV curing. Previous work reported that the optimum amount of photoinitiators for Irgacure 500 and Darocur 1173 were 2% and 1.5%, respectively [13].

This optimum amount of both photoinitiators improved the abrasion resistance of coatings.

Sample Formulations

Table 1 shows that VTMOS coupling agent, Irgacure 500 and Darocur 1173 photoinitiators loading were constant for all formulations. 4-hydroxy anisole stabilizer loading was constant for all formulations except for Formulation 13 (F13) and Formulation 14 (F14). The concentration of maleic anhydride in water was also constant except for Formulation 1 (F1), Formulation 3 (F3), and Formulation 6 (F6).

F1 was prepared without Aerosil OX-50 whereas 10% Aerosil OX-50 was added into Formulation 2 (F2) in order to study the effect of Aerosil OX-50 on the coatings. According to unpublished work by Salleh (2002), the significant improvement in scratch resistance was only achieved at 10% or more Aerosil OX-50 loading.

30% TPGDA in F1 and F2 was replaced with 30% PETIA in Formulation 3 (F3) and Formulation 4 (F4). However, F4 was added with

10% Aerosil OX-50. Both monomers, 17.5% TPGDA and 17.5% PETIA, and 10% Aerosil OX-50 were added in Formulation 5 (F5). The effect of Aerosil OX-50 and the different types of monomer on the hardness coating were investigated in these formulations.

Ebecryl 600 prepolymer and 30% PETIA monomer were used in Formulation 6 (F6) to Formulation 12 (F12). The Aerosil OX-50 loading in F6 to F12 was increased from 0 to 30%. These F6–F12 formulations were formulated in order to study the effect of Aerosil OX-50 loading on the viscosity of uncured formulations and finished coatings.

Similar to F6 to F12, Ebecryl 600 prepolymer and Aerosil OX-50 were also used in Formulation 13 (F13) and Formulation 14 (F14). However, 15% TPGDA and 15% PETIA monomers were used simultaneously in F13 and F14 instead of 30% PETIA in F6 to F12. PETIA amount was decreased from 30% in F12 to 15% in the F13 and F14. The F14 has the highest Aerosil OX-50 loading (35%) compared to the rest of formulations (F1–F13). As the Aerosil OX-50 loading increased in F13 and F14, the filler loading time during the synthesizing process also increased. As a result, the use of 4-hydroxy anisole stabilizer had been increased from 480 mg for F12 to 720 mg for F13 and F14 in order to maintain the stability of F13 and F14. The stability of the formulation decreased with increasing the loading time due to the increase of time taken for the formulations to undergo the shearing action of rotating blades and the heat exposure. In this study, the reduction of stability means that the formulation either gelled immediately after the synthesis or has a shorter shelf life. To maintain the stability of F13 and F14, the temperature of synthesis also decreased from 65 °C for F1 to F12 to 60 °C for F13 and F14.

All prepolymers, monomers, and filler were weighed in each plastic container, respectively. Meanwhile, VTMOS coupling agent was weighed in an amber glass bottle. All materials were kept closed and stored away from light. Both maleic anhydride and 4-hydroxy anisole were ground to powder from their original pebble-like state. Meanwhile, distilled water and maleic anhydride were weighed in a same bottle. The sealed bottle was then immersed in an Elma Transsonic T1040/H sonic water bath for about 45 min to obtain a homogenous solution. The ultrasonic sound produced vibrations that contained energy. This vibration caused molecules to collide with each other and facilitated the solution process. The molecular collisions, in turn, produced heat that increased the solubility of maleic anhydride in water. After the process, no more powder particles were present, indicating a homogenous solution had been formed. The bottle was then stored inside a dark cabinet.

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Mixing

The mixing process was done using the Dispermat mixer VMA-Getzmann GmbH. Neslab USA RTE-211 water bath was used to regulate the temperature of the mixing container. The water bath was allowed to stabilize at 65°C prior to the mixing process. The Dispermat mixing blades with 70 mm radius were chosen, to suit the size of the double wall stainless steel container.

Ebecryl 230 prepolymer was poured into the mixing container. Care was taken to ensure that it did not spill to the side of the container or onto the shaft of the mixing blades. The rotation speed for the mixing blades was increased gradually in order to prevent the prepolymer from spilling out of the container. The monomer TPGDA was added to the container, followed by 4-hydroxy anisole. TPGDA reduced the viscosity of the prepolymer and facilitated the mixing process.

The maleic anhydride solution was then added followed by dropwise addition of VTMOS. It is worth noting that during the addition, the rotation speed of the blade mixer should be increased slowly. The duration of VTMOS addition was within 30 min. Finally, the silica nanofiller was added. The mixture was allowed to mix thoroughly for an hour at 2500 rpm. After mixing, the mixture was poured into an amber glass bottle and immediately brought to the ultrasonic probe.

The ultrasonic probe was dipped into the center of the bottle. The purpose was to remove air bubbles produced during the addition and mixing process. Air bubbles scatter the light, causing the mixture to appear milky. When the ultrasonic probe stopped, the clarity of the mixture increased after air bubbles were removed. The bottle was left for at least one hour for the mixture to stabilize. The bottle was then sealed and kept in a dark cabinet.

Some content from the bottle was put into another smaller one. Then the photoinitiators were weighed based on the weight of the content in the small bottle. The photoinitiators were also weighed in the same bottle. The photoinitiators and the content of the small bottle were mixed together. The percentage of photoinitiators used is shown in Table 1. The content of the small bottle with both photoinitiators was then stirred at 200 rpm for one hour using Heidolph RZR-2000. For EB-cured coatings, the use of photoinitiators was omitted.

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Coating and Curing Process

The coating process was similar for both UV and EB curing methods; only the curing process was different. The K Paint automatic film applicator was switched on. The perforated metal base of film applicator was vacuumized to hold the cover paper and the glass plate properly. The cover paper was used to prevent the coating materials from dripping into the vacuum holes. The slit thickness of the applicator bar used was 150 microns. The thickness of 150 microns was chosen because the coating was easier to peel for gel content measurement and reduced the possibilities of torn samples during the abrasion tests. The glass plate was put on the cover paper and the applicator bar was fixed and locked. Then the coating was poured on the glass plate and the coating process was started.

For UV curing, the coated glass was then brought to the UV machine and was put on the conveyor. The UV lamps of the IST UV curing machine had been switched on and stabilized for 15 min prior to the curing process. The conveyor speed of the curing machine was set at 10 meters/min. At this speed, this machine produced 0.150 J/cm^2 of energy. The first coated glass was allowed for one pass under the UV light. The number of passes was 2, 3, 4, 5, 8, 10, and 12 each for the next seven coated glass plates.

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For EB curing, the same formulation without photoinitiators was coated on a glass plate and subjected to electron beam curing using Curetron made by Nissin High Voltage. The dosage provided by the machine is 10 kGy (kiloGray) for each pass. The number of passes was also 2, 3, 4, 5, 8, 10, and 12 each for the next seven coated glass plates.

The purpose of the highest 12 passes (UV curing) and 120 kGy (EB curing) was to determine whether the polymerization process reached the plateau. After the irradiation, the glass plates were taken out and conditioned at 25°C under laboratory ambience for 4 h before measuring their pendulum hardness and gel content. The pendulum hardness and gel content tests were done to find the optimum curing doses for each UV and EB curing. After these dosages were determined, the whole process of coating and curing was repeated to prepare the samples for scratch and abrasion resistance measurement.

Characterization of Nanocomposite Coatings

Byk Pendulum Hardness Tester was used to measure the surface hardness of nanocomposite coatings according to DIN 53157, by adapting the König method. After hardness measurement, the coatings were peeled off and placed into the Soxhlet Extractor for 16 h to measure their gel contents. Results from pendulum hardness and gel content were used to determine the suitable dosages for UV/EB curing. The thumb twist method was also used to support the pendulum hardness and gel content results. The selected dosages were then used to cure coatings for scratch and abrasion resistance tests. 150 μm-thick coatings were cured on PVC plates and decorative furniture papers for scratch and abrasion resistance tests, respectively. The scratch resistance of a coating film was measured using Erichsen Scratch Tester Model 413 according to DIN 53799. Two types of scratching needles (tip) were used, stainless steel ball tip and diamond tip. The higher the value (Newton) obtained, the better the scratch resistance. Taber Abraser 5151 was used to measure the abrasion resistance of the cured coatings according to DIN 68861.2. The testing was done at 50 wheel rotations with 500 g load for each abrasive wheels. In the case for abrasion resistance, the lower the weight loss, the better.

RESULTS AND DISCUSSION

The Effect of Radiation Dosages

The surface hardness is an important property of coatings, because harder surfaces resist the scratch and abrasive forces better. Figures 1 and 2 show the effect of radiation dosages on the hardness of coatings cured by UV and EB, respectively. Surface hardness increased with increasing radiation dosages (number of passes) for all samples. This was due to the increase in crosslinking with increasing radiation dosages. Within the experimental parameters, a plateau was not observed. It is highly possible that a plateau will be observed if the radiation dosages were to increase. However, it was

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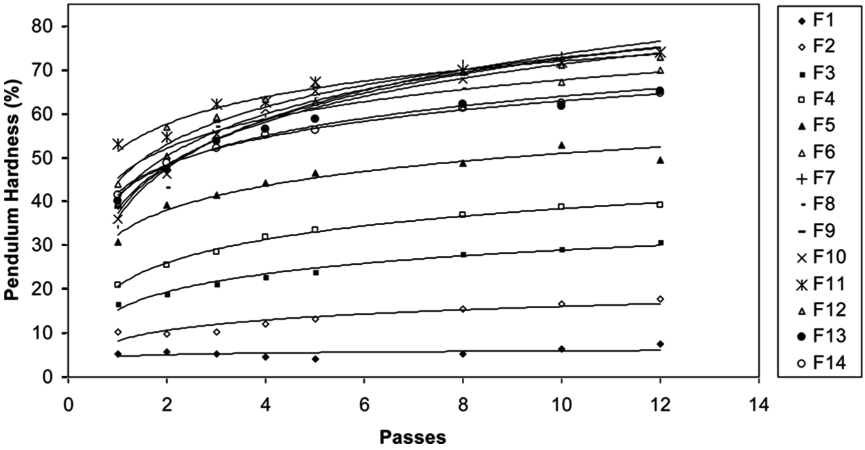

FIGURE 1 The effect of radiation dosages on the pendulum hardness of coatings cured by UV radiation.

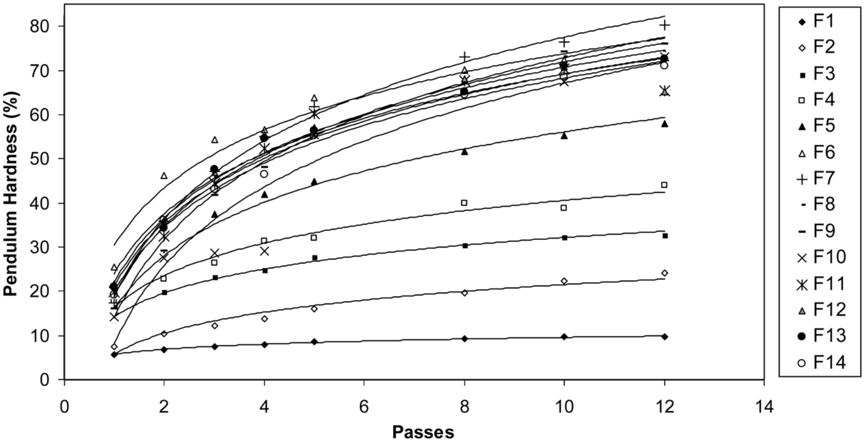

FIGURE 2 The effect of radiation dosages on the hardness of coatings cured by EB radiation.

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not economical to do so. Gel content measurement was done later, because it measured the overall cure rather than surface cure. According to Ali et al. [14], film (pendulum) hardness represented the crosslinking density at the film surface, whereas the gel content represented the crosslinking density present in the entire film.

Figure 3 shows that increase of radiation dosages increased the gel content of UV cured coatings. From the figure, the optimum curing dosage was 5 passes (as indicated by arrow). This is because for most formulations (F1–F6, F13, and F14), no significant increase in gel content was observed with increasing radiation dosages. Economically, it would be a disadvantage to increase dosages which does not result in significant improvement in gel content. Unfortunately, the measurement of gel content for EB-cured samples was very difficult to do because they were hard and brittle. EB-cured coatings adhered strongly to the substrate, suggesting some grafting had been formed. This is not surprising, because with EB radiation, electrons penetrating into the substrate were able to generate radicals. Radicals from the substrate combined with radicals from the network, led to improved adhesion by grafting at the substrate/coating interface. A similar theory was also proposed by Mehnert et al. [15]. Any attempt to peel the coatings from the glass plates proved futile because the coatings broke into small fragments. These fragments escaped through the wire mesh during the solvent extraction. Thus, the gel content measurement could not be made.

In order to solve this problem, the thumb twist method was used to find the suitable curing dosage for EB-cured coatings. The thumb was

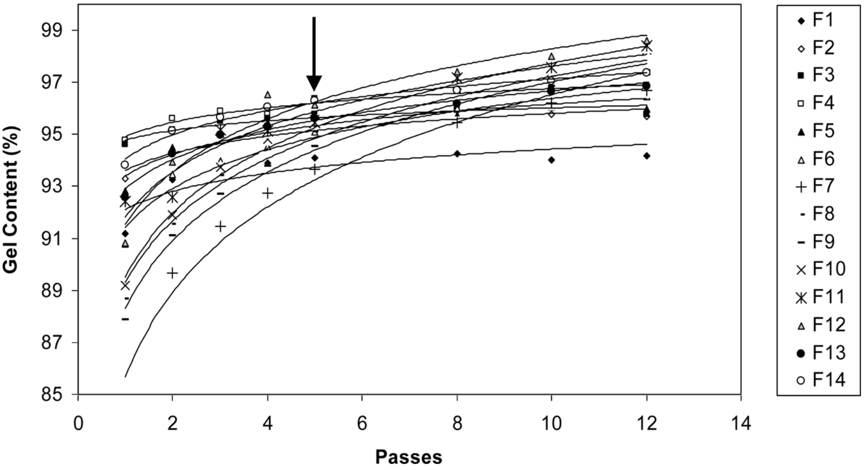

FIGURE 3 The effect of radiation dosages on the gel content of coatings cured by UV radiation.

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firmly pressed to the cured film and twisted. According to Lowe [16], any deformation of the coating film or remaining tackiness of the coating or transfer of the coating film to the thumb was classified as a failure. It was found that at 100 kGy, the coating was already non-tacky, except for Formulations 1 and 2. These two formulations appeared tacky even when cured at 120 kGy or 12 passes under UV light. This was possibly due to the nature of the formulations. Kumar et al. [9] also reported similar findings, with an even higher dosage (350 kGy). UV-cured coatings were also found to be non-tacky from 5 passes onwards, in accordance with the gel content results. These values (5 passes and 100 kGy) were used to cure coatings to be tested for scratch and abrasion resistance.

With increasing radiation dosages, the surface hardness of the samples was found to increase for both types of radiation techniques, i.e., UV and EB. After the first pass under the UV light, the photoinitiators dissociated and produced active radicals. These radicals attacked the unsaturated double bond in the acrylate prepolymers and monomers, thus initiating the crosslinking process. More passes under UV light produced more crosslinking and subsequently higher gel content. For formulations cured by EB, the electron beam attacked and removed the electron from certain atoms and initiated the crosslinking process. More passes under the EB also produced more crosslinking within the coatings and yielded higher gel content.

The Effect of Types of Radiations

Figures 1 and 2 show that the pendulum hardness values of UV-cured coatings were higher than EB-cured coatings in the initial passes before the reversal of trend afterwards. This was due to the difference in chemistry of UV and EB curing. With UV, the concentration of free radicals at the surface was high, unlike EB where it was low. With EB, the radical concentration increased inward, from the surface to substrate. This was partly because the wet film decreased the energy of the electrons, thereby slowing them so that they would undergo mechanisms to initiate cure. A similar theory was suggested by Webster [2]. So, it would be safe to say that UV-cured coatings cured from outside to inside (surface would harden first), whereas its EB counterparts cured thoroughly the other way round (subsurface would harden first).

From Figures 4 to 8, the effect of UV/EB radiation on the pendulum hardness of the coatings can be observed. In Figure 4, F6 without nanosilica filler was taken as an example. UV coatings were harder until 5 passes before being overtaken by EB coatings. After 5 passes, the EB-cured coatings were much harder than UV coatings. The next 15% increase in nanosilica content of F9 (Figure 5) showed a surprising trend. The intersection was moved forward. The trend continued until F12 with 30% nanosilica (Figure 6). In Figure 6, UV cured F12 coatings were always harder than EB-cured F12.

It could be suggested that the increase in nanosilica content influenced the curing behavior (radical polymerization) of UV coatings.

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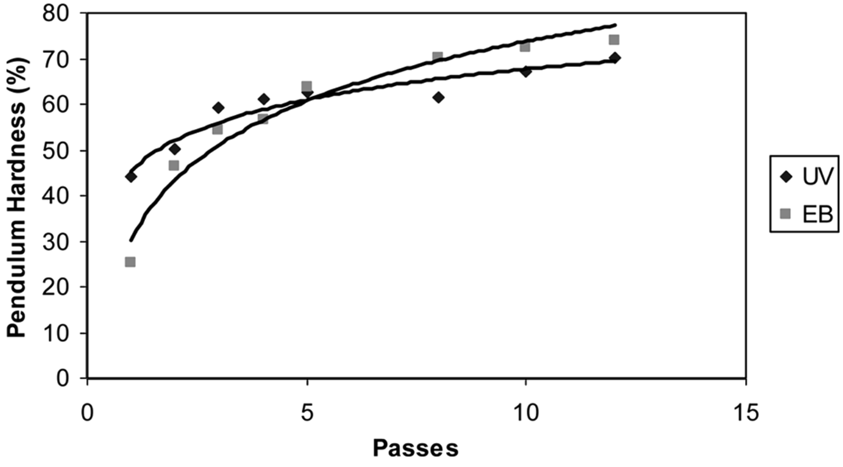

FIGURE 4 The effect of UV/EB radiation on the pendulum hardness of F6.

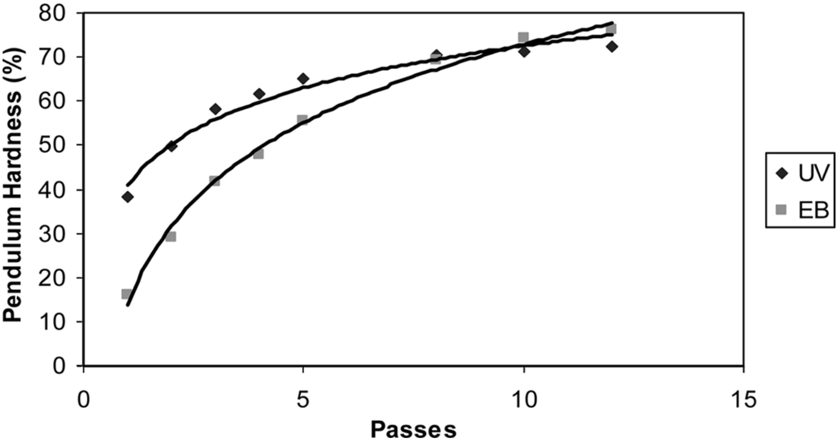

FIGURE 5 The effect of UV/EB radiation on the pendulum hardness of F9.

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The silica nanoparticles apparently accelerated the cure reaction and cure rate of the UV-curable acrylate system, most probably due to the synergistic effect of silica nanoparticles during the photopolymerization process. A study by Cho et al. [17] also reported similar findings. From F12 to F13, the curing trend changed drastically (Figures 6 and 7). This was proof that the curing behavior was also influenced by the temperature and formulation. Comparison between F13 (Figure 7) and F14 (Figure 8) shows that the intersection shifted

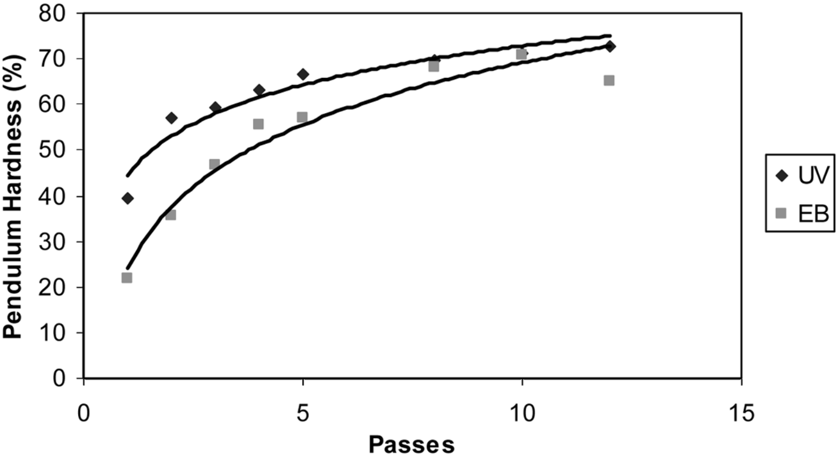

FIGURE 6 The effect of UV/EB radiation on the pendulum hardness of F12.

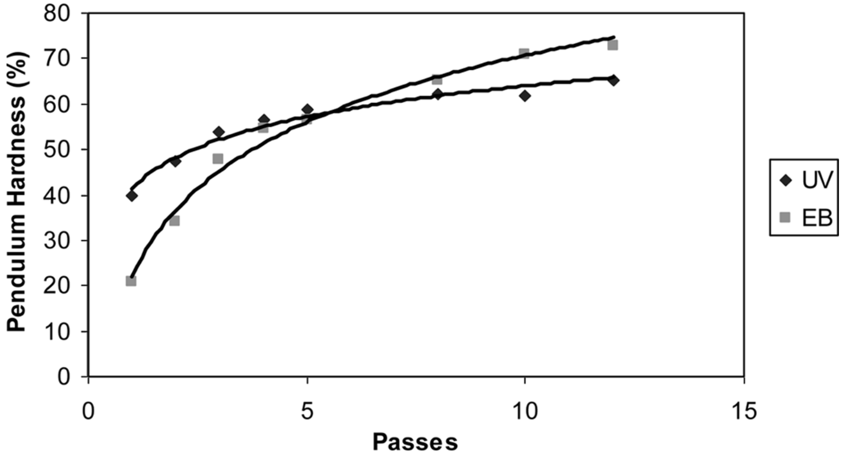

FIGURE 7 The effect of UV/EB radiation on the pendulum hardness of F13.

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to the right, albeit slightly. This confirmed that even though the formulations were not changed, the amount of silica nanoparticles still influenced the cure reaction and cure rate of the UV-curable acrylate system.

For the purpose of discussion, the results of scratch resistance by diamond tip were used (Table 2). It is clear that EB curing produced coatings with higher scratch resistance. This was due to the fact that the EB curing process produced a more uniform polymeric film. Work by Ruiz et al. [11] also reported similar findings. In other words,

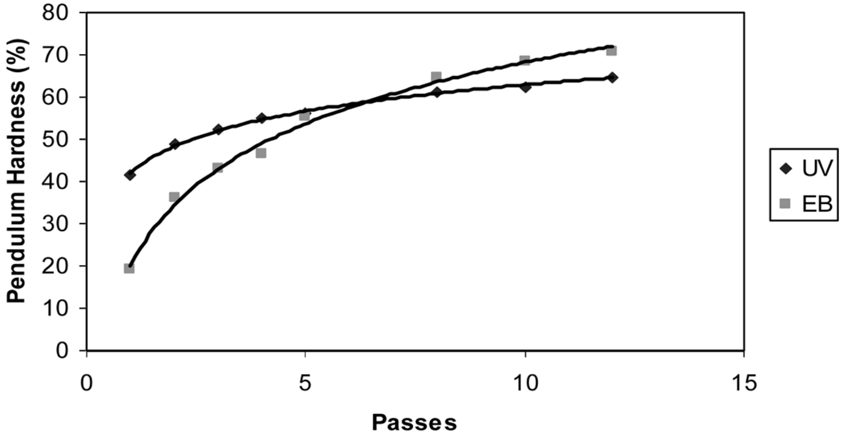

FIGURE 8 The effect of UV/EB radiation on the pendulum hardness of F14.

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TABLE 2 Scratch Resistance of the Surface Coatings with Film Thickness of 150 μm

Formulation	UV-curing Steel ball diameter 1 mm (N)	UV-curing Diamond tip 90° (N)	EB-curing Steel ball diameter 1 mm (N)	EB-curing Diamond tip 90° (N)
F1	≤0.1	0.2	≤0.1	0.2
F2	0.1	0.3	0.1	0.4
F3	1	0.2	0.4	0.2
F4	2	0.3	0.5	0.4
F5	8	0.6	7	0.5
F6	>10	0.7	9	0.9
F7	>10	0.8	>10	1
F8	>10	1.0	>10	1.5
F9	>10	1.5	>10	2.5
F10	>10	2	>10	3.5
F11	>10	2.5	>10	4
F12	>10	2.5	>10	4.5
F13	>10	3	>10	3
F14	>10	3	>10	3.5

EB coatings were hard from inside to the surface. The hard surface and subsurface provided some resistance to the scratching by diamond tip. For UV-cured coatings, even though their surfaces hardened quickly, they were softer than EB-cured coatings. Although measurement of scratch resistance by steel ball was also done, the limitation of the steel ball was obvious, since it could not measure the actual hardness of coatings with Ebecryl 600 (F6–F14). Nevertheless, the results from steel ball tip were useful in confirming that the scratch resistance was also increased with the addition of nanosilica.

Figure 9 shows the effect of radiation types on the abrasion resistance of the coatings. According to Kumar et al. [9], abrasion resistance is one of the most complex properties to achieve, as it is both a surface as well as subsurface property. Almost all coatings cured by UV showed higher resistance to abrasive forces compared to those cured by EB. F1 was not included in the graph since they were already torn at 20 rotations. Still, UV-cured F1 lost only 11.4 mg compared to EB-cured F1 which lost 26.8 mg. Even though UV coatings cured from outside in, the curing was not uniform compared to EB-cured coatings. The surface might be hard since the concentration of radicals was highest at the surface, but below the surface (subsurface), the coatings were flexible. When the abrasion test was done, the flexible subsurface provided some resistance to the abrasive rotations by clogging the

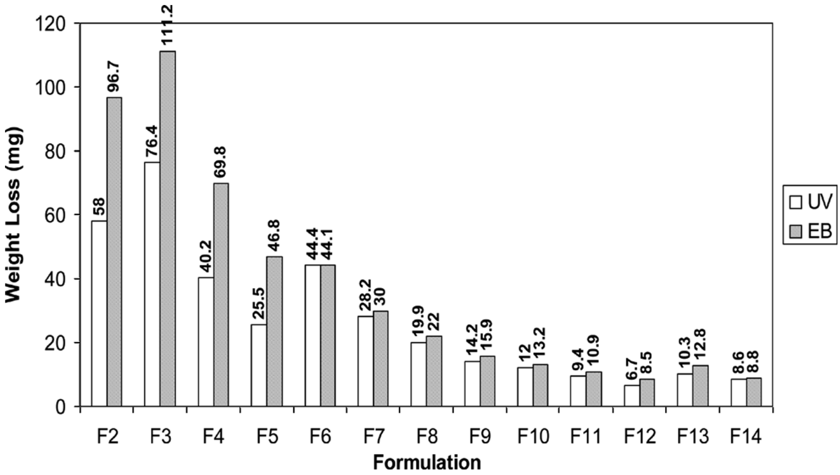

FIGURE 9 The abrasion resistance of surface coatings with film thickness of 150 μm.

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abrasive wheels. Meanwhile, EB-cured coatings, with uniform hardness from inside to surface, performed poorly in abrasion tests. This was attributed to the fact that EB-cured coatings contained higher degrees of branching and crosslink densities than comparable UV ones. Hard and brittle EB-cured coatings would be abraded much more easily and turned to dust by abrasive rotations.

CONCLUSION

With increasing radiation dosages, the surface hardness of the samples was found to increase for both types of radiation techniques, i.e., UV and EB. EB-cured coatings were found to have better adherence to substrate. This was possibly due to grafting between both interfaces formed during EB irradiation. The increase in nanosilica content was found to accelerate the cure reaction and cure rate of the UV-curable acrylate system. As a result, the coating hardness was also increased. Application-wise, UV curing technology is more suitable for producing coatings with better abrasion resistance, whereas EB curing technology is capable of increasing the scratch resistance of the coatings. This was due to the way both radiations cured a coating. UV coatings cured from outside to inside whereas for EB coatings, it was vice versa. UV coatings had a flexible subsurface which resisted abrasion. Meanwhile, EB coatings were thoroughly cured and hard which could resist the scratching.

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