Effect of Fillers on Curing of Wood Surfaces with Polyester Oligomers by Ultraviolet Radiation

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ABSTRACT: A set of formulations was prepared with polyester acrylate (oligoester M-9050) oligomers in combination with reactive diluents of different functionalities such as ethylhexyl acrylate, tripropylene glycol diacrylate , and trimethylol propane triacrylate (TMPTA). The thin films were prepared with these formulated solutions under UV radiation on a glass plate, and their physical properties such as pendulum hardness and gel content were studied. The formulation containing TMPTA showed the greatest pendulum hardness and gel content. The polished wood surfaces were cured with these formulated solutions. Physical properties such as pendulum hardness gloss at 20° and 60° angles, adhesion, abrasion resistance, and scratch hardness of UV-cured surfaces of the wood were characterized. The formulation containing TMPTA had the best physical properties. Two types of filler, sand and talc, were used in the base coat to obtain these better properties. Both fillers improved the properties; however, the 1% sand– and 4% talc–containing formulations performed better. © 2003 Wiley Periodicals, Inc. J Appl Polym Sci 89: 3826–3834, 2003

Key words: surface coating; UV curing; wood; polyester acrylate; fillers

INTRODUCTION

Photopolymerization initiated by ultraviolet (UV) radiation is receiving considerable attention for rapid and solvent-free curing of polymer films. This solvent-free polymerization proceeds rapidly at room temperature with a fraction of the energy requirements of conventionally (thermally) cured systems. The recent surge in applications of UV-initiated photopolymerization has been motivated by at least two factors: environmental concerns about the production of volatile organic solvent emissions and the need for high-speed reaction to enhance production rates with a high-quality end product. As a result, UV-initiated photopolymerization is finding application in a variety of areas, including curing of coating inks, adhesives, and electronics.[1–4]

Wood is a principal construction material all over the world. Its availability in various forms and sizes, together with such properties as relatively great strength with respect to weight, ease of shaping, fastening, and low heat conductivity have made it an outstanding building material from the time of the first settlers to the present. Even today, despite extensive use of other structural materials, the railroads; highway departments; telephone, telegraph, electric light, agricultural, building, and navigation industries; and producers of household articles such as bedsteads, chairs, tables, beams, and pillars for cottages and huts depend on wood. But all the inherent properties of wood are not favorable for diverse uses; some characteristics hinder its versatile and potential applications. The hygroscopic nature of wood is significantly responsible for the dimensional instability with variation in moisture content that eventually results in weeping and uneven distortion. Wood's nonabrasive character and resistance to weather conditions of wood are relatively low. Under some conditions that are favorable to them, wood bores, termites, bacteria, insects, microbes, and fungi can destroy wooden structures in a short period of time, and wood is susceptible to combustion as well. To overcome these drawbacks of wood, it is necessary to modify the wood or wood surface. The total supply from these trees can not satisfy the growing demand for fine-quality timbers from around the world. Therefore, trees in forests are being indiscriminately destroyed to satisfy this demand for wood. As a result, the global forest reserve is being rapidly depleted fast. It is difficult to procure teak, a very high-quality timber, for the market, unlike a few years back. Therefore, people are using low-grade wood as a substitute. But this type of wood does not produce the desired product quality. The purpose of the present study was to

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TABLE I
Composition of Different Formulations (%)

Formulation	Oligomer (M-9050)	Monomer			Photo initiator IRG-651	$CaCO_3$	Sand
		EHA	TPGDA	TMPTA			
A1	50	48			2		
A2	50		48		2		
A3	50			48	2		
A4	50	24	24		2		
A5	50	24		24	2		
A6	35		63		2		
A7	20		78		2		
A8	35			63	2		
A9	20			78	2		
A10	49			48	2	1	
A11	48			48	2	2	
A12	47			48	2	3	
A13	46			48	2	4	
A14	45			48	2	5	
A15	49			48	2		1
A16	48			48	2		2
A17	47			48	2		3
A18	46			48	2		4
A19	45			48	2		5

improve the surface of simul, a low-quality fuel wood of Bangladesh through curing a polyester acrylate coating in combination with reactive monomers of different functionalities by ultraviolet (UV) radiation. The effect of fillers such as sand and talc on the physical properties of cured wood surface was also studied.

EXPERIMENTAL

Materials

A polyester-acrylated oligoester (M-9050) was procured from Radcure (Drogenbos, Belgium). The photoinitiator, Irgacure 651 (UV 200–450 nm absorption band) was procured from Ciba-Geigy (Switzerland). The reactive monomer diluents, 2-ethylhexyl acrylate (EHA), tripropylene glycol diacrylate (TPGDA), trimethylol propane triacrylate (TMPTA), and calcium carbonate ($CaCO_3$) were obtained from Merck (Germany). Sand (SiO_2) was collected from the local market. Simul (*Salmalia malabarica*), a low-quality wood mainly used for making matchsticks, was collected from the local market and used as the substrate for coating.

Method

A set of formulations were prepared with a polyester acrylate (M-9050) oligomer with other additives, as shown in Table I. These formulations were used to prepare thin polymer films on glass plates as well as on the wood substrate. These films were characterized. The wood surface was thoroughly polished with suitable sandpaper (Nos. 1 and 0). The substrate (wood or glass plate, $10 \times 5 \times 0.2$ cm) was coated with the formulated solution using bar coater No. 0.18 from Abbey Chemicals Co. (Australia). The average thickness of the film was 36 ± 3 μm, which was measured by a Digimatic micrometer (Mitutoyo, Japan). The substrate was passed several times under a UV lamp (254–313 nm, 2 kW intensity) from IST Technik (Model U-200-M-Tr, Germany) to ensure curing on the substrate. The speed of the conveyor was 4 m/min. The film hardness of the UV-cured film was determined while the film was still on the substrate. Several techniques were adopted to measure film hardness. Pendulum hardness of UV-cured films was directly measured with the help of a pendulum hardness tester (Model 5854; BYK, Labotron, Germany). Scratch resistance, Bochhloz resistance, and macro- and microhardness were determined using a universal hardness tester (Model 43/E, Germany). Indent depth, length, and adhesion of the films were determined with the help of a PIG universal tester (Model 3410; BYK, Labotron, Germany). Adhesion strength and the chipped-off area of the curedcoated film were determined by an adhesion tester (Model 525, ASTM Standard D 4541, Erichsen, Germany). The abrasion resistance of UV-cured wood surface was determined by an Abraser (Model 5130, Erichsen GmbH &CO. KG, Germany). These results are expressed as wear factor or Taber wear index (which is the loss of weight in milligrams per thousand cycles of abrasion). The gloss of the UV-cured film on the wood surface was measured at 60° and 20° angles using a gloss meter (Model Sheen 155, Germany).

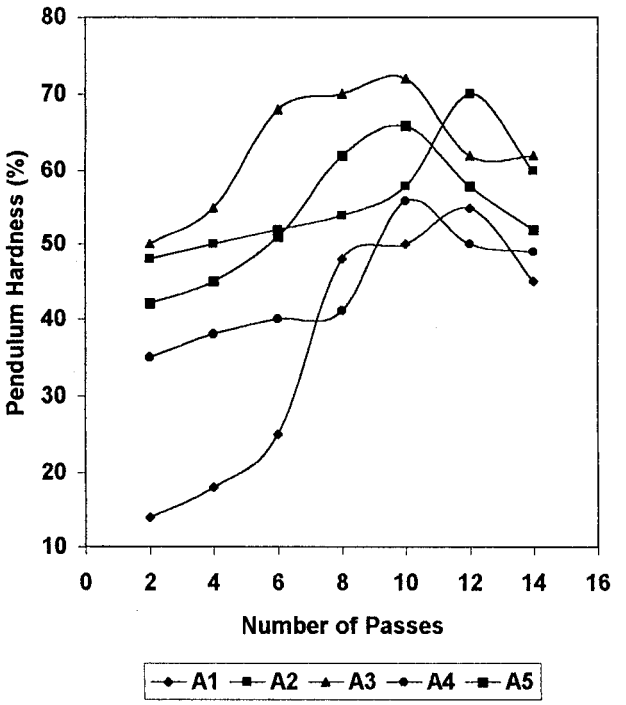

Figure 1 Pendulum hardness of UV-cured films against number of passes.

RESULTS AND DISCUSSION

It was necessary to characterize the UV-cured polymer films before measuring various properties of the UV-cured coating on the wood surface. Most of the data in this report are averaged values of at least five samples, and the results obtained are within ±2%.

Characterization of film

Pendulum hardness

The surface hardness of the UV-cured thin polymeric films prepared on the glass plate was determined by the pendulum method. Results for pendulum hardness (PH) of the films against different UV doses, represented by the number of passes, are shown graphically in Figure 1. The hardness increased with an increasing number of passes. This may indicate that the crosslinking density increased with an increasing dose. After attainment of maxima, the hardness decreased with an increasing dose. This may indicate that the polymer films prepared at the radiation dose of maxima started to degrade at higher doses.[5]

However, for formulations A1–A5, the highest value of pendulum hardness (PH), 82%, was obtained with A3 at 12 passes, which contained 48% TMPTA. The second-highest value (75%) was obtained with A2, which contained 48% TPGDA, a difunctional monomer. The physical properties of the UV-cured films depended on the functionality of the monomers as well as their glass-transition temperature (T_g).[6] The T_g values of TMPTA, TPGDA, and EHA were 250°C, 190°C, and −50°C, respectively.[7] TMPTA is a triacrylate monomer that contains three acrylated reactive sites through which a quick polymerization network is accomplished.[8] Monomer TMPTA had the highest glass-transition temperature (T_g = 250°C) among all the diluents used and thus was conductive, yielding a stronger film surface.[6] Obviously, the films of the diluents with low T_g values should have registered a low hardness. Actually, this can be observed in Figure 1, which shows that EHA (T_g = −50°C) films produced the lowest hardness because it is a low-T_g monomer. TPGDA (T_g = 90°C) produced moderate PH values, compare to TMPTA and EHA, because it is a difunctional monomer with a higher T_g value than that of EHA and a lower one than that of TMPTA.

Gel content

The amount of gel content, the crosslinking density of UV-cured films, is plotted in Figure 2 against the number of passes. With an increase in UV radiation the amount of gel content of the polymer films increased. Maximum gel content was achieved at different radiation doses depending on the nature of the formulations. The maximum gel value lay between the 8th and 10th passes. From formulations A1–A5, formulations A3 produced the highest gel (96%) at the eighth pass, which contained 48% TMPTA. After attainment of maxima at a radiation dose, the value of gel content decreased at higher dose. This may be

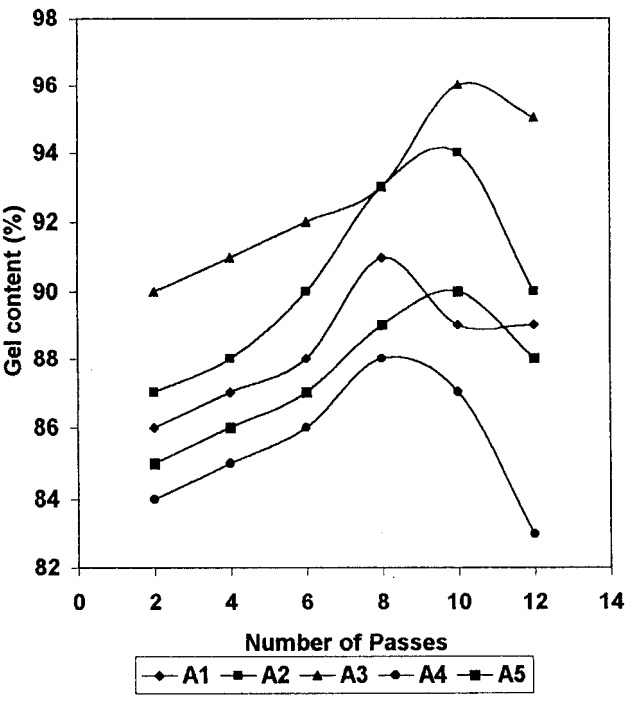

Figure 2 Gel content of UV-cured films against number of passes.

caused by the degradation of the polymer film at a higher dose.

Selection of base- and top-coat formulations for application on wood surfaces

Because the highest value of pendulum hardness (82%) and gel content (96%) was achieved by formulation A3, it was selected as the base coat for application on a wood surface because hard film is required for coating on a wood surface. Then formulations A6–A9 were developed for the application on wood surface as top coats.

Application on wood surface

Having characterized the UV-cured polymer films, the different formulations were applied on polished wood surfaces. Base coats were partially cured on the wood surface with UV radiation. The substrates were polished with sandpaper (No. 0) before final coating with the top coat. Coatings were cured on the wood surface with different numbers of passes under a UV lamp. Different physical parameters such as macroscratch hardness, pendulum hardness, Taber index, microgloss, and adhesion strength were determined as a function of radiation intensity expressed by the number of passes.

Pendulum hardness

Shown in Figure 3 is the plot of pendulum hardness of the UV-cured wood surface against number of passes. Pendulum hardness increased with an increase in UV dose, represented by number of passes. This reflects an increase in crosslinking density in the polymer with an increase in UV dose. It can be observed from Figure 3 that the maximum PH values were attained between the 8th and the 10th passes. The highest hardness (59%) was achieved with coating by formulation c (A3 + A8), where A3 and A8 are the base coat and top coat, respectively. Formulation A8 contained the highest amount of TMPTA, which is a very good crosslinking agent because of its branchlike structure with three acrylated functional groups. Coating *b* (A3 + A7) and *d* (A3 + A9) produced lower PH values because these formulations contained smaller amounts of oligomer (M-9050) than the other formulations.

Macroscratch hardness

The macroscratch hardness (MSH) of the UV-cured coating on a wood surface was measured in terms of load (weight) in grams required to manifest a scratch on the surface of the coatings. The greater the index of macroscratch hardness, the better is the coating. From Table II it can be observed that coating *c* (A3 + A8) possessed a higher macroscratch hardness (300), where A3 is the base coat and A8 the top coat. The microscratch hardness of a coated wood surface could be measured, but the micro values for all the formulations were out of range. The indent length is the width of the scratch. If the indent length is small, it indicates a better cohesion among the polymer matrix. Thus, the smaller the width, the better is the surface coating in hardness. Thus, it was observed that the width decreased with increasing hardness, which is related to the curing of the surface, that is, the crosslinking density on the surface. Similarly, the indent depth, that is, the depth of scratch yield was expected to be smaller for better film. The fully cured film will have less penetration during the scratch. It is apparent from Table II that macroscratch hardness and Buchholz resistance (of coatings a, b, c, and d) increased with an increasing number of passes, whereas indent length and indent depth decreased and coating *c* (A3 + A8) was better with respect to indent depth and indent length.

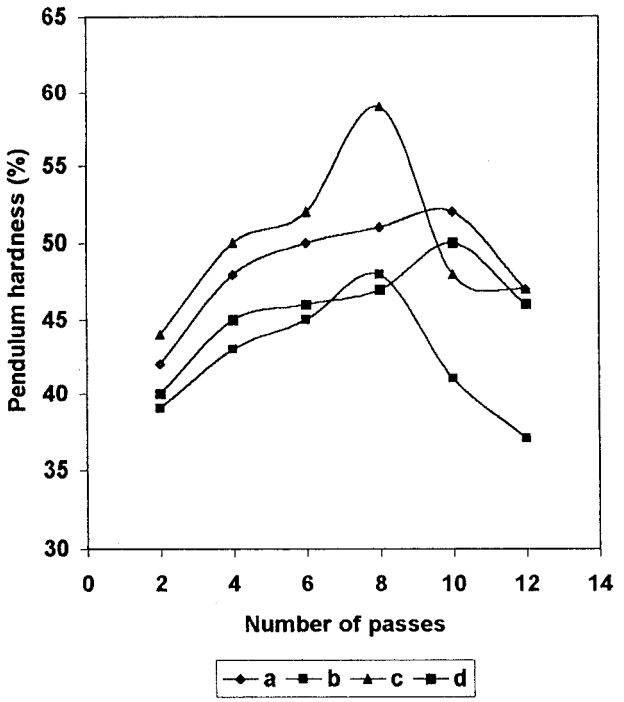

Figure 3 Pendulum hardness of UV-cured wood surface against number of passes.

Adhesion strength

Adhesion of UV-cured coating on the wood surface was measured by both the crosscut and pulling-off methods. It can be observed from Table II that the chipped-off area decreased with an increasing dose. The coating of formulation c (A3 + A8) adhered well with the substrate, and when using the crosscut technique, much less of the area was chipped off. In Figure

TABLE II
Different Physical Properties of UV-Cured Coating on Wood Surface

Properties	No. of passes	Base coat + topcoat A3 + A6 (= a)	A3 + A7 (= b)	A3 + A8 (= c)	A3 + A9 (= d)
Macro scratch hardness	2	200	180	200	190
	4	210	200	220	195
	6	220	210	250	200
	8	250	240	250	240
	10	250	240	260	245
	12	260	250	300	260
Indent length (mm)	2	0.85	0.90	0.88	0.90
	4	0.82	0.85	0.85	0.88
	6	0.80	0.80	0.70	0.85
	8	0.79	0.75	0.70	0.80
	10	0.77	0.73	0.68	075
	12	0.75	0.72	0.65	0.70
Indent depth (μm)	2	6	7	7	7
	4	5	6	6	6
	6	5	5	<5	6
	8	<5	<5	<5	5
	10	<5	<5	<5	<5
	12	<5	<5	<5	<5
Indent Buchholz resistance	2	118	111	111	111
	4	125	118	118	118
	6	125	125	>125	118
	8	>125	>125	>125	>125
	10	>125	>125	>125	>125
	12	>125	>125	>125	>125
Adhesion (% chipped-off area)	2	5	5	2	5
	4	4	5	2	5
	6	4	5	0	4
	8	0	4	0	4
	10	0	2	0	0
	12	0	2	0	0

4 adhesion strength (according to the pull-off method) for coatings a, b, c, and d is plotted against number of passes. It was observed that the value increased with the number of passes for all formulations. The greatest strength required to pull off the coating of formulation c (A3 + A8) from a wood surface was 0.9 N/mm^2.

Abrasion resistance

The Taber index of UV-cured coating on a wood surface by formulations a–d is plotted in Figure 5 versus the number of passes. A lower Taber index value indicates higher abrasion resistance. The Taber index decreased with an increasing number of passes. The lowest value of the Taber index found (545) was that offormulation *c* (A3 + A8).

Microgloss

Quality of a surface coating can be roughly estimated by visual observation. Gloss is the reflection of light from the coating at a certain angle with the vision. Normally, the gloss of coating is determined at two angles, such as 20° and 60°. The results of gloss esti-

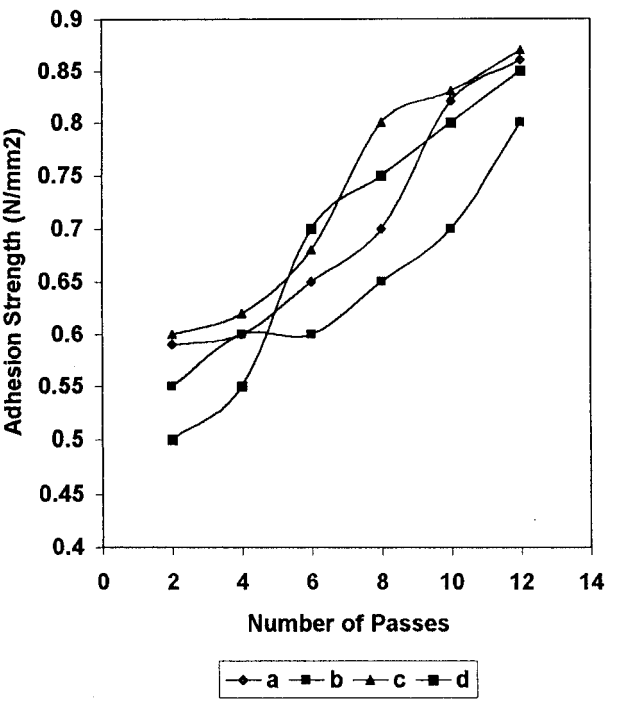

Figure 4 Adhesion strength of UV-cured films on wood surface against number of passes.

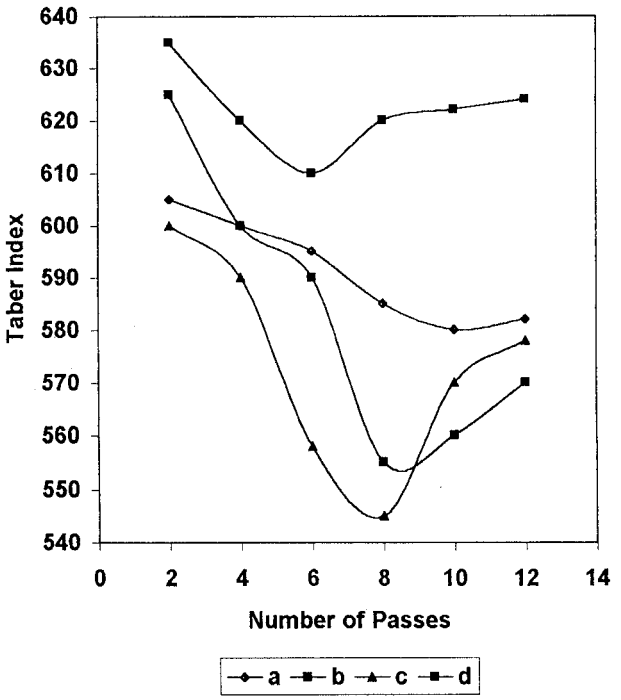

Figure 5 Taber index of UV coating on wood surface against number of passes.

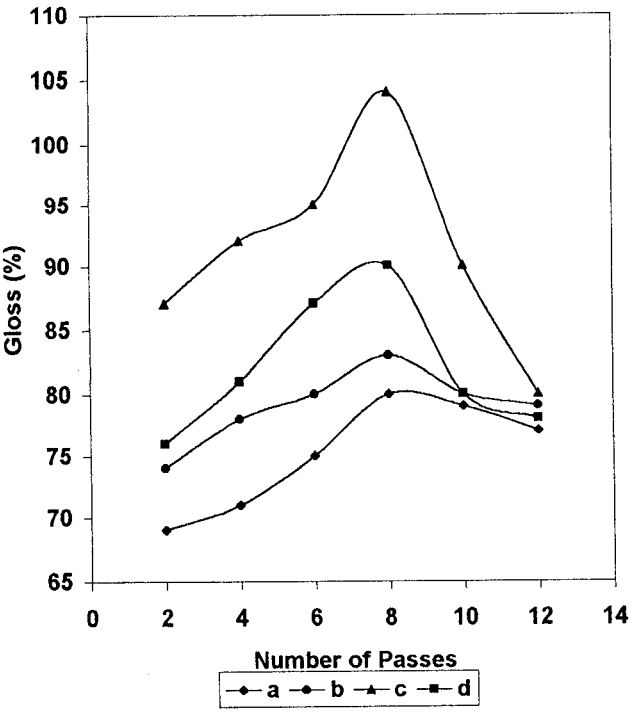

Figure 7 Microgloss (at 60°) of UV-cured coating on wood surface against number of passes.

mated on UV-cured coating on a wood surface against number of passes are shown in Figures 6 and 7 for 20° and 60° angles, respectively. It can be observed that the value of gloss increased with curing of the coating, which is represented by the number of passes under the UV lamp. Figures 6 and 7 indicate that the highest gloss at angles 20° and 60° was observed for coating c (A3 + A8). It was also observed that after attainment of its maximum value, the value of gloss decreased with any further increase in the dose of radiation.

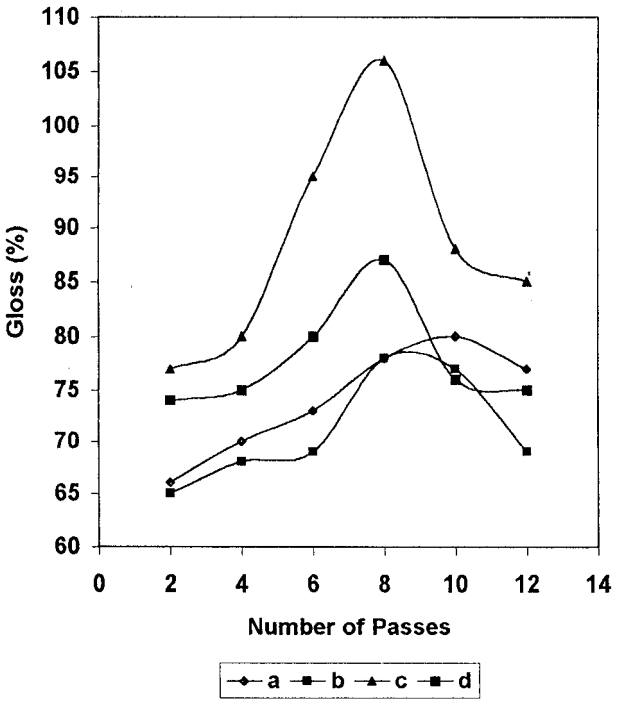

Figure 6 Microgloss (at 20°) of UV-cured coating on wood surface against number of passes.

Effect of fillers

To study the effect of fillers, formulations A10–A14, which contained 1%–5% talc, and A15–A19, which contained 1%–5% sand, were prepared. These formulations were used as base coat for modification of the wood surface where formulation A8 was used as the top coat.

Effect of $CaCO_3$

It can be observed from Table III and Figure 8 that the value of pendulum hardness and adhesion strength of coating increased up to 1%–4% talc in the base coat. Gloss (%) was virtually unchanged up to 0%–1% $CaCO_3$, then it decreased. The highest PH (74%) was yielded by the formulation containing 4% $CaCO_3$ in the base coat, whereas the lowest PH (59%) was found for the formulation that did not contain talc in the base coat. The increase in PH value from the addition of $CaCO_3$ probably occurred because $CaCO_3$ is the most widely used filler and extender pigment in the plastics industry, and it can be compounded easily with epoxy resin. This type of filler reduces the void space of the

TABLE III
Various Properties of UV-Cured Coating on Wood Surface Containing Talc and Sand in Base Coat

Properties	No. of passes	Base coat + topcoat									
		A10 + A8 (= e)	A11 + A8 (= f)	A12 + A8 (= g)	A13 + A8 (= h)	A14 + A8 (= i)	A15 + A8 (= j)	A16 + A8 (= k)	A17 + A8 (= l)	A18 + A8 (= m)	A19 + A8 (= n)
Macroscratch hardness	2	250	250	275	275	260	255	260	250	245	200
	4	260	265	280	275	280	260	270	260	250	225
	6	270	270	285	290	280	280	290	265	255	240
	8	285	290	285	295	300	300	300	270	260	245
	10	300	300	310	310	300	300	310	280	270	250
	12	300	300	310	320	300	310	300	280	265	250
Indent length (mm)	2	0.90	0.85	0.85	0.80	0.85	0.85	0.80	0.90	1.00	1.05
	4	0.85	0.85	0.80	0.80	0.80	0.85	0.78	0.85	0.90	1.00
	6	0.70	0.70	0.70	0.70	0.65	0.80	0.70	0.85	0.85	0.95
	8	0.70	0.70	0.65	0.65	0.65	0.75	0.65	0.80	0.80	0.85
	10	0.70	0.65	0.60	0.60	0.63	0.70	0.60	0.75	0.75	0.80
	12	0.70	0.65	0.60	0.60	0.62	0.60	0.60	0.70	0.7	0.70
Indent depth (μm)	2	7	6	6	5	6	6	5	7	9	10
	4	6	6	5	5	5	6	<5	6	7	9
	6	5	<5	<5	<5	<5	5	<5	6	6	8
	8	<5	<5	<5	<5	<5	<5	<5	5	5	6
	10	<5	<5	<5	<5	<5	<5	<5	<5	<5	5
	12	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
Indent Buchholz resistance	2	111	118	118	125	118	118	125	111	100	95
	4	118	118	125	125	125	118	>125	118	111	100
	6	125	>125	>125	>125	125	125	>125	118	118	105
	8	>125	>125	>125	>125	>125	>125	>125	125	125	118
	10	>125	>125	>125	>125	>125	>125	>125	>125	>125	125
	12	>125	>125	>125	>125	>125	>125	>125	>125	>125	>125
Adhesion (% chipped-off area)	2	6	6	5	4	5	5	4	6	8	10
	4	5	4	5	4	5	5	4	5	5	6
	6	4	4	0	0	2	4	2	4	5	5
	8	2	0	0	0	0	0	0	3	4	3
	10	2	0	0	0	0	0	0	0	2	3
	12	0	0	0	0	0	0	0	0	0	0
Adhesion (N/mm^2)	2	0.60	0.70	0.80	0.80	0.40	0.50	0.45	0.45	0.43	0.40
	4	0.60	0.75	0.81	1.00	0.60	0.75	0.70	0.50	0.45	0.45
	6	0.70	0.80	0.90	1.10	0.75	0.80	0.80	0.60	0.55	0.50
	8	0.75	0.90	1.00	1.40	0.80	0.90	0.85	0.70	0.60	0.60
	10	0.90	1.30	1.30	1.50	0.70	0.95	0.90	0.65	0.70	0.50
	12	0.80	1.20	1.20	1.60	0.60	0.90	0.87	0.55	065	0.50
Macroscratch hardness	2	250	250	275	275	260	255	260	250	245	200
	4	260	265	280	275	280	260	270	260	250	225
	6	270	270	285	290	280	280	290	265	255	240
	8	285	290	285	295	300	300	300	270	260	245
	10	300	300	310	310	300	300	310	280	270	250
	12	300	300	310	320	300	310	300	280	265	250

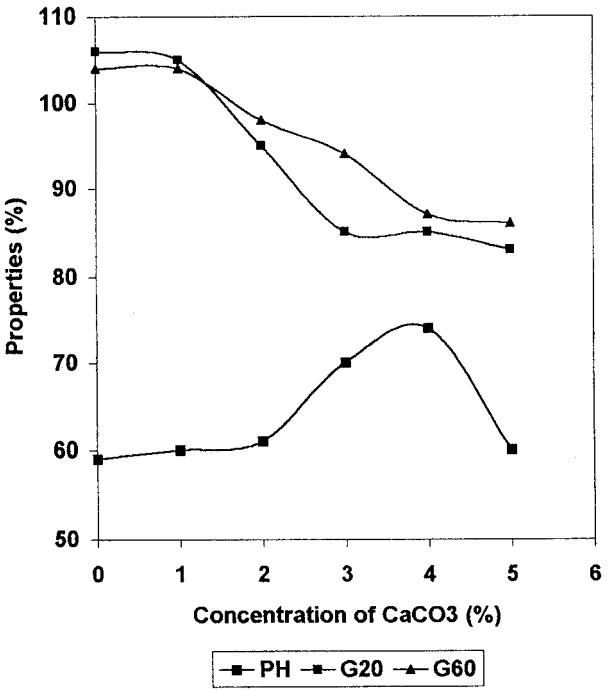

Figure 8 Combine properties of UV-cured coating on wood surface against concentration of calcium carbonate in base coat.

surface and perhaps increases the hardness of the coated surface, such as micro- and macroscratch hardnesses (MSH) of the coating, which are defined in terms of the load weight in grams required to manifest a scratch on the surface of the coatings. More weight is required if the resistance to scratch is higher because of better physical properties. Thus, the greater the index of MSH, the better are the coatings. Microscratch hardness for all the coatings was found to be out of range. The MSH values are shown in Table III. The coating that contained 4% $CaCO_3$ in the base coat showed the highest MSH (320). It can be observed from Figure 8 that as the amount of $CaCO_3$ content increased, gloss values at both angles also decreased to the same extent because $CaCO_3$ probably prevents the crosslinking process, and $CaCO_3$ may act as a free-radical scavenger because of its inherent chemical nature. The highest surface gloss (106% and 104%) at both angles was obtained from the coating that was free of $CaCO_3$ filler, followed by the formulations with 1% filler in the base coat. According to the results of the Taber index, shown in Table III, for an increasing filler ($CaCO_3$) concentration in the base coat up to 4%, the resistance toward abrasion wear also increased to the same extent; the index value increased more with greater filler content, than with that of the lowest value. The adhesion strength required to pull out the coatings from the surface are shown in Table III. With respect to $CaCO_3$ content, it was found that adhesion (N/mm^2) decreased up to 3% $CaCO_3$ and slightly increased for 4% and 5% $CaCO_3$ in the base coat.

Effect of sand

It can be observed from Table III and Figure 9 that the value of pendulum hardness and adhesion strength of the coating increased up to 1%–2% sand in the base coat. Gloss decreased by increasing the percentage of sand in the base coat. It can be observed from Figure 9 that as the concentration of sand increased from 0% to 3%, the values of pendulum hardness also increased, and after that the PH value decreased with increasing sand content. The increase of PH with the addition of sand is most probably resulted from the adhesion properties and from the monomer–sand–oligomer bonds behaving just like coupling agents. The decrease occurred because the sands tookk part in the crosslinking reaction. The highest PH (79%) was obtained from the coatings that contained 3% sand and the lowest from the coating whose sand content was 1%. The gloss decreased slightly at the angle 60° with sand content up to 3%, but gloss reduction was high for 4% and 5% sand. At the 20° angle the formulation with a sand content of 1% in the base coat yielded more gloss than did the formulation with no sand content in its base coat. With higher contents of sand used as fillers, the gloss at 20° also decreased. The results of MSH, abrasion resistance, and adhesion strength (in N/mm^2) tests are shown in Table III. It was observed that the highest MSH, 310, was obtained from the coatings that contained 2% sand in the base coat. Also, the highest adhesion value (0.95 N/mm^2) was achieved by the formulation containing 2% sand in the base coat.

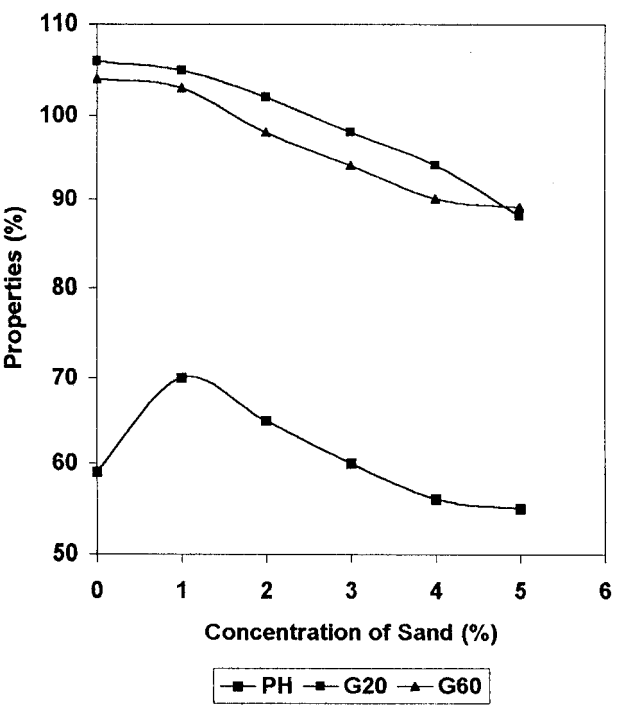

Figure 9 Combine properties of UV-cured coating on wood surface against concentration of sand in base coat.

CONCLUSIONS

UV-cured polymer films and wood surface coatings were obtained with different formulations made with polyester acrylate, EHA, TPGDA, TMPTA, and fillers (sand and talc). After characterization of these films, formulation A3 (which showed best hardness and gel content) was selected as the base coat. Characterization of the coatings revealed that coating with formulation c (A3 + A8) offered the best physical and mechanical properties. Both fillers (sand and talc) improved all properties except microgloss. So 1% sand or 4% talc can be used in a formulation to improve surface properties. It can be concluded that after coating, simul can be used in some cases with high-density quality wood.

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