Effect of $CaCO_3$ on the Performance of Partex Surface Modification by Ultraviolet Radiation Curing Method

M. MIZANUR RAHMAN,[1,2] MUBARAK A. KHAN,[1] M. KHABIR UDDIN,[1] K. M. IDRISS ALI,[1] A. I. MUSTAFA[1,2]

[1] Radiation and Polymer Chemistry Laboratory, Institute of Nuclear Science and Technology, Bangladesh Atomic Energy Commission, P.O. Box 3787, Dhaka, Bangladesh

[2] Department of Applied Chemistry and Chemical Technology, University of Dhaka, Dhaka-1000, Bangladesh

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ABSTRACT: Several UV-curable formulations containing epoxydiacrylate (EB-600) oligomer with a tri-functional monomer, trimethylol propane triacrylate (TMPTA), and photoinitiator Irgcure-369 were developed to improve the surface of Partex. Filler or extender $CaCO_3$ was incorporated into the solution. Thin polymer films were prepared on glass plate with these formulated solutions and finally applied on polished Partex surface, and both were cured under UV-radiation. The properties of UV-cured thin films were studied as a function of $CaCO_3$ concentration. Pendulum hardness and gel content were found to decrease on glass plate with the increase of $CaCO_3$ concentration. Pendulum hardness, scratch hardness, and abrasion resistance of the cured Partex were found to be higher with the increase of $CaCO_3$ content up to 4%. Thus, the formulation containing 4% $CaCO_3$ showed the best performance over all formulations containing $CaCO_3$. © 2001 John Wiley & Sons, Inc. J Appl Polym Sci 81: 1858–1867, 2001

Key words: Partex; surface modification; UV curing; $CaCO_3$ filler; UV–radiation

INTRODUCTION

Particle board is a well-known construction material. It is used for subflooring as core stock for veneered furniture and decorative panels. It is replacing plywood and lumber in some structural applications.[1] However, the manufacturing cost of particle board is high in the context of Bangladesh. But Partex produced in Bangladesh using jute stick and urea formaldehyde resin by a compression method can be used as a substitute to particle board. The main raw material for Partex manufacture (jute stick) is abundantly available in Bangladesh. However, Partex has some drawbacks; all the inherent properties of Partex are not favorable for diversive uses; and some of them restricts its versatile and potential applications. Partex is hygroscopic, and is dimensionally instable with variation of moisture content. This eventually results in weeping and uneven distortion. The nonabrasive character and the resistance to weather conditions of Partex are relatively low. Under some favorable conditions termites, bacteria, insects, microbes, and fungi can destroy Partex structure in a short period of time, and Partex is susceptible to combustion as well. Some work has already been done[2–4] to improve low-grade wood (simul) surface by UV-cured epoxy coatings. It is envisaged that the coating of polymer on the Partex surface can similarly be improved through UV radiation. Tangential strength can be enhanced to the same degree as the longitudinal strength (the real unilateral strength of Partex). The coating makes the surface more resistant to

Correspondence to: M. A. Khan.

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abrasion, biological attack, and weather conditions. It also improves its electrical and thermal insulating capabilities for probable use as insulators. Property of the cured surface depends on the adhesive force as well as the extent of crosslinking. This force is enhanced by the presence of an OH group in the substrate and monomer unit. Calcium carbonate ($CaCO_3$) is the most widely used filler or extender pigment in the plastic industry. Polyvinyl chloride, polyolefines, phenolics, polyesters, and epoxies are all resins with which $CaCO_3$ can be compounded. Perhaps no other area has generated more interest in expanding the use of fillers in plastic than the technology of chemical coatings and coupling agents. Coated $CaCO_3$ is currently available in finer form. Most of the commercial products are coated with steric acid or calcium stearate. Coating leads to improved filler dispersion and superior rheological and physical properties. The present study is related to the modification and characterization of Partex surface coated with epoxy (EB-600) mixed with TMPTA (trimethylol propane triacrylate) containing $CaCO_3$ (1–5%) and cured under UV radiation.

EXPERIMENTAL

Materials

Ebcryl-600, a diacrylated epoxy oligomer and trimethylol propane triacrylate (TMPTA), a trifunctional monomer were procured from Radcure (Drogenbos, Belgium). Filler $CaCO_3$ is a product of BDH Ltd. Methyl Ethyl Ketone (MEK) was used as an antibubbling agent, and was obtained from Merck (Germany). Irgacure-369 (IRG-369) has an absorption band of 200–450 nm, and was used as photoinitiator during the UV radiation process to initiate photochemical reactions. It was procured form Ciba-Geigy. Partex was collected from the local market of Bangladesh.

Methods

Different formulations were developed with Ebcryl-600 (EB-600) in combination with reactive monomer. TMPTA in the presence of filler $CaCO_3$, antibubbling agent MEK and photoinitiator IRG-369 in the proportions, as mentioned in Table I. Thin polymer films were prepared under UV radiation by coating a glass plate (3″ × 2″) with these formulated solutions using a bar coater (No. 0.0018 of Abbey Chemicals Co., Australia). This produced films of 36 ± 3 μm thickness on the plate. A UV minicure machine (IST Tecknik Germany, Model Me-200-UV) was used to cure the film on the plate (as well as on the Partex surface) using a UV lamp (254–313 nm, 2KW). The substrate was run under the lamp at a speed of 4 m/min with the help of a conveyor belt interlocked with the UV radiation system. The minicure has the efficiency of ±1%.

Table I Composition of Different Formulations (% w/w)

	Formulations						
Chemicals	R1	R5	R6	R7	R8	R9	R10
EB-600	50	18	49	49	49	49	49
TMPTA	48	79	48	47	46	45	44
$CaCO_3$	—	—	1	2	3	4	5
IRG-369	2	2	2	2	2	2	2
MEK	—	1	—	—	—	—	—

Physical Properties of the Cured Polymer Films

Film hardness was directly measured by the pendulum method[5] using a Pendulum hardness (PH) tester (model 5458, BYKE, Labotron). After 24 h of UV radiation, the cured film was used for hardness measurement when the UV-cured film was still on the glass plate. PH of the cured Partex surface was also determined by the same method. Gel content was determined by extracting a known weight of the cured film with hot benzene for 48 h in a soxhelt. For this purpose, the film was wrapped in a stainless steel net that was put into the soxhelt. The loss of weight of the cured film due to extraction process yields the gel content. It was calculated using % Gel = 100 − 100 $(w_0 - w_e)/w_0$, where w_0, is the weight of the cured film before extraction and w_e is weight of the extracted film after drying at 105°C until a constant weight was achieved. Macro- and micro-scratch hardness was measured by a Universal Hardness Tester (model 413/E, Erichsen, Germany).

Application on Partex Surface

The Partex sample was cut into small pieces (size 4″ × 4″ × 1/6″) and dried in an oven at 105°C to remove the free moisture from the Partex until constant weight. The surface of the Partex samples were polished smoothly with the help of a suitable sand paper (No. 1 and 0). Then it was coated first with a base coat using a drawn down bar (No. 0.0028) and cured by a single pass under the UV lamp. It was polished again and coated with a top coat using the bar No. 0.0018, and finally the coating was cured under UV light at different intensities (number of passes). Scratch resistance, Buchhloz resistance, macro- and microscratch hardness of cured films were determined. Indent length, depth, and adhesion in terms of percent chipped off area of the films were determined with the help of PIG Universal Tester (model 3410, BYK, Labotron, Germany). Surface gloss of the modified Partex was measured at two angles 60° and 20° using a Microgloss Meter (Sheen-155) from Sheen Co., UK. Abrasion wear of the coated Partex surface was measured by the Taber abrasion method using a Taber Abraser (model 5130 of Richness Co., Germany). The lower Taber index indicates better resistance to abrasion by the cured film. The adhesion ability of the UV-cured film was determined by measuring the force that was applied to peel a certain portion of the cured film with the help of an adhesion tester (model 525) from Erichsen Co., Germany. The treated Partex substrate was subjected to severe weathering testing by simulating sunlight (4 h) using a UV lamp (313 nm) and condensation (2 h); the accelerated weathering testing was carried out for 500 h with the help of a tester (Q-UV-26200, Q-Panel Co., USA). The loss of PH, gloss, adhesion, and MSH as a result of the weathering test was determined.

RESULTS AND DISCUSSION

It is necessary to characterize UV-cured polymer films before applying to the surface of Partex. Most of the data presented in this report are averaged values of at least six different results.

Characterization of Polymer Films

Film Hardness

Surface hardness, an index of crosslinking density at the surface of UV-cured thin polymer films prepared on a glass plate was determined by the pendulum method. Results of pendulum hardness (PH) of the films prepared at different UV doses represented by the number of passes are graphically shown in Figure 1. It is observed that the hardness (PH) of the films prepared with different

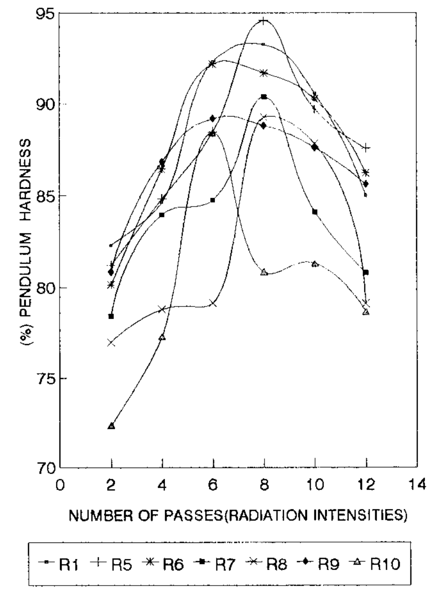

Figure 1 Pendulum hardness of UV-cured polymer films against number of passes (radiation intensities).

formulations increases with radiation dose intensity. The process of curing continues with UV radiation up to a certain UV dose, at which it gains the maximum hardness—some at sixth pass, while others at the eighth pass. After attaining the maximum hardness, the PH value then decreases. This means that degradation of polymer occurs at higher doses.[6] The highest PH value (94.57%) is given at the eighth pass by the formulation R5 containing no $CaCO_3$. The second highest PH value (93.26%) is imparted by R1 followed by R6 (92.17%), containing 1% $CaCO_3$. The lowest PH value is given by the formulation R10, containing 5% $CaCO_3$. As the content of $CaCO_3$ increases into the formulations the PH value decreases, because $CaCO_3$ may prevent the crosslinking process. $CaCO_3$ may act as a free radical scavenger due to its inherent chemical nature.

Gel Content

The gel content is a representation of crosslinking density through the entire films. Figure 2 shows the gel content of polymer films against the number of passes. The maximum gel content value (99.65%) is achieved by formulation R5, because it contains more TMPTA (79%) (Table I), and TMPTA has trifunctional acrylated groups that have a branching effect[7] and can yield more crosslinking in the cured film. A multifunctional vinyl monomer promotes rapid free radical propagation reaction[8] leading to network (crosslinking) structures. When TMPTA concentration is increased, the amount of residual unsaturation is also increased as a consequence of faster rate of formation of the three-dimensional network, causing restricted mobility at an early stage. The crosslinking rate, especially during the early stage of radiation, is proportional to the TMPTA concentration.[9] The second highest gel content is obtained by the formulation R1 (97.98%) followed by R6, R7, R8, R9, and R10. The maximum gel content varies between 99.65 to 93.78%. This is a sign of very good crosslinking phenomena among the various constituents present in different formulations. For formulations containing 1 to 5% $CaCO_3$, the gel values gradually decrease from R6 to R10 as the $CaCO_3$ content increases. However, these values, which are still quite high, indicate high crosslinking ability under UV radiation.

Macroscratch Hardness (MSH) of the Films

A macroscratch hardness test of the cured film was performed while the films were still on the glass plate. However, the maximum values obtained for PH, gel, and MSH in each of these formulations are plotted in Figure 3 to have a comparative look at a glance in respect of different properties of the formulations. The highest MSH value (370 g) was obtained by the formulation R1 containing no $CaCO_3$ followed by the formulation R6, R7, R8, R9, and R10 and R5, respectively. Thus, as the concentration of $CaCO_3$ increases with the oligomer and the monomer; MSH on the glass plate decreases; the values also decrease as the concentration of TMPTA decreases.

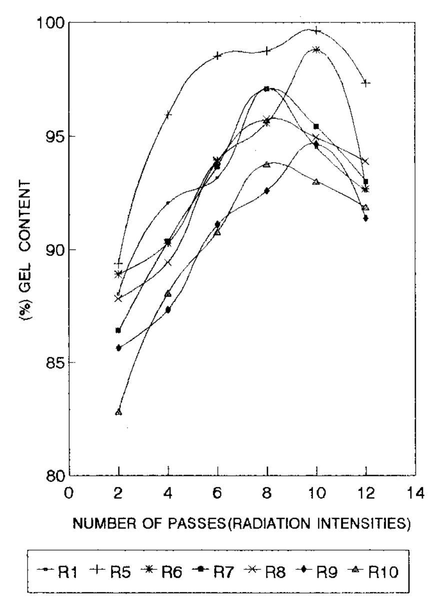

Figure 2 Gel content of UV-cured polymer films against number of pass (radiation intensities).

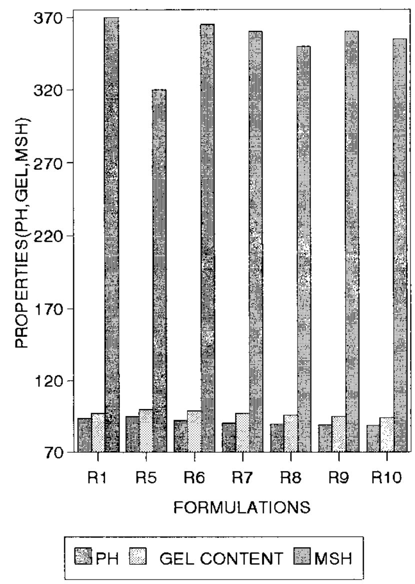

Figure 3 Various physical properties of UV cured polymer film against different formulations.

Application on the Partex Surface and Determination of the Effect of $CaCO_3$ on the Partex Surface Modification

After characterization of the UV-cured polymer films, different formulations were applied on the Partex surface. Formulation R1, R6, R7, R8, R9, and R10 were arbitrarily chosen as base coats due to their higher viscosity and higher hardness on glass plate, whereas R5 was chosen as the top coat because of its lower viscosity and its antibubbling property. The base coats were applied on polished Partex surface and partially cured with UV radiation. Then the substrates were polished with suitable sandpaper (No. 0). Top coat was applied on Partex surface and cured with UV radiation at different number of passes viz. 4, 6, 7, 8, 10, and 12. Various physical and mechanical parameters, particularly macroscratch hardness, indent length, indent depth, Buchhloz resistance, film adhesion strength, and percent chipped off area were determined as a function number of passes. The results are shown in Table II.

Surface Hardness

Macroscratch hardness of the coating is measured in terms of load (weight) in gm required to man-

Table II Various Physical and Mechanical Properties of UV-Cured Surface Coatings on Partex

Properties	No. of Passes	D = (R1 + R5) Base Coat R1 + Top Coat R5	E = (R6 + R5) Base Coat R6 + Top Coat R5	F = (R7 + R5) Base Coat R7 + Top Coat R5	G = (R8 + R5) Base Coat R8 + Top Coat R5	H = (R9 + R5) Base Coat R9 + Top Coat R5	I = (R10 + R5) Base Coat R10 + Top Coat R5
Taber wear index (abrasion resistance)	4	578	597	681	421	504	497
	6	469	672	825	390	201	302
	7	432	667	830	452	112	219
	8	403	593	793	186	169	209
	10	462	777	770	567	174	199
	12	452	704	557	356	171	212
Adhesion (N/mm^2)	4	1.50	1.25	0.70	1.05	0.75	1.10
	6	1.75	1.25	1.10	1.00	1.15	1.05
	7	1.65	1.50	1.05	0.75	1.30	1.10
	8	1.45	1.00	0.75	1.00	1.45	1.15
	10	1.25	10.5	0.95	0.75	1.05	1.20
	12	1.05	1.45	0.85	1.00	0.85	1.40
Macro Scratch Hardness (g)	4	630	640	625	640	700	680
	6	650	645	675	690	710	7000
	7	690	690	720	735	740	720
	8	715	720	730	750	770	740
	10	745	730	735	775	785	775
	12	735	735	745	765	775	750
Hardness indent length (mm)	4	0.85	0.75	0.85	0.70	0.75	0.80
	6	0.71	0.75	0.80	0.75	0.65	0.80
	7	0.74	0.80	0.75	0.75	0.70	0.85
	8	0.60	0.80	0.75	0.75	0.65	0.85
	10	0.63	0.85	0.85	0.80	0.70	0.85
	12	0.80	0.85	0.80	0.70	0.75	0.80
Indent depth (μm)	4	6	<5	6	<5	<5	5
	6	<5	<5	5	<5	<5	5
	7	<5	5	<5	<5	<5	6
	8	<5	5	<5	<5	<5	6
	10	<5	6	6	5	<5	6
	12	5	6	5	<5	<5	5
Indent Buchhloz resistance	4	118	>125	118	>125	>125	125
	6	>125	>125	125	>125	>125	125
	7	>125	125	>125	>125	>125	118
	8	>125	125	>125	>125	>125	118
	10	>125	118	118	125	>125	118
	12	125	118	125	>125	>125	125
Adhesion (% Chipped off area)	4	0	4	0	4	0	4
	6	0	0	0	0	0	2
	7	0	0	0	0	0	0
	8	0	2	0	0	0	0
	10	0	4	2	0	0	2
	12	2	0	2	2	0	2

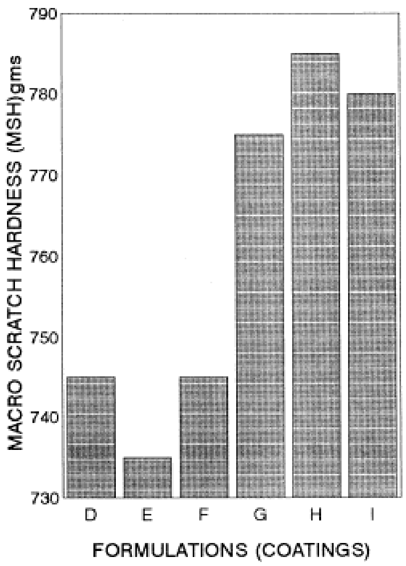

Figure 4 Macro scratch hardness of UV-cured surface coatings on partex against different formulations.

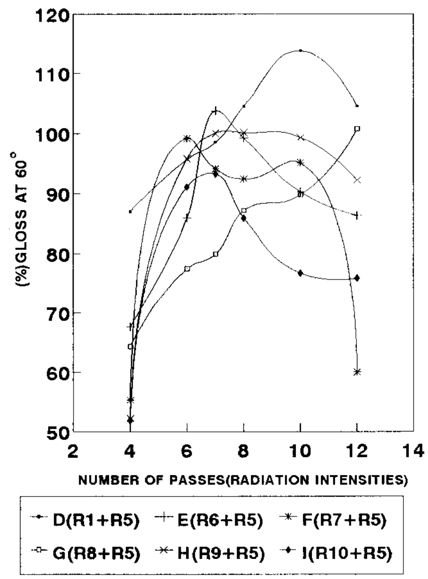

Figure 5 Micro gloss properties at 60° angle of UV-cured surface coatings on partex at different UV dose.

ifest a scratch on the surface of the coatings. More weight is needed if the resistance to scratch is higher due to its better physical properties. Thus, the more the index of macroscratches hardness the better the coating. The values of MSH are plotted in Figure 4 against the formulations. Highest value (785 g) is given by the formulation H (R9 + R5) and the lowest (735 g) by the formulation E (R6 + R5) (Table II). As the concentration of $CaCO_3$ increases from 0 to 4%, the values of MSH increases; but for 5% $CaCO_3$, the values MSH decreases.

The microscratch hardness test carried out for the coating was found to be out of range for all samples, indicating that better hardness was found where $CaCO_3$ is included with the formulation. The indent length is small; this indicates that there is better cohesion among formulation H (R9 + R5), and thus, induces the smallest indent length on the coating. Similarly, indent depth is depth of the scratch. The least of serated depth of the film represents its better physical properties.

Surface Gloss

Gloss is another important property for Partex surface coatings. The gloss is generally measured at two angles (60° and 20°) of the plane of the modified surface. Results of gloss are shown in Figure 5 (60°) and Figure 6 (20°). Gloss determined at the 60° angle is generally higher than at a 20° angle. Gloss is related to the extent of curing.[2] Thus, the gloss increases with an increase of crosslink density. From the figure it is clear that as the concentration $CaCO_3$ increases in the base coat from 1 to 5% the gloss of the modified surface also decreases at both 60° and 20° angles. The coating E (R6 + R5) containing 1% $CaCO_3$ gives the highest gloss both at 60° (103.75%) and 20° (90.30%) at the seventh pass in each case. The

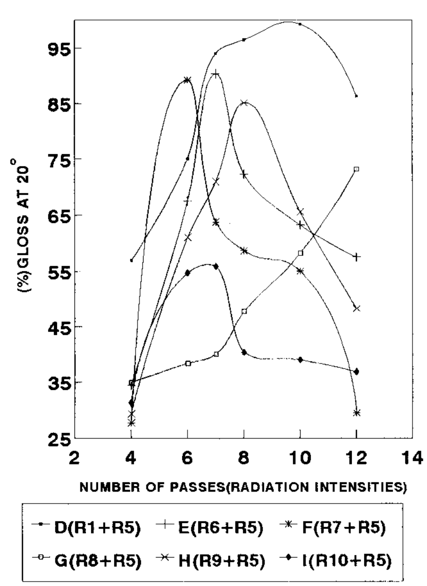

Figure 6 Microgloss properties at 20° angle of UV-cured surface coatings on partex at different UV dose.

lowest gloss value at 60° and 20° as observed are 93.26 and 55.80%, respectively, and are obtained with formulation I (R10 + R5) containing 5% $CaCO_3$ in the base coat.

Pendulum Hardness

Surface hardness of the film coating measured by Pendulum technique is plotted against number of passes in Figure 7. The coating E (R6 + R5) with base coat R6 having 1% $CaCO_3$ yielded the highest PH, which is greater than that with no $CaCO_3$ in the base coat, i.e., D (R1 + R5). The highest PH value (76.72%) for E (R6 + R5) is obtained at the sixth pass, whereas the highest PH value (70.17%) for D (R1 + R5) is obtained at the eighth pass. From the figure it is found that if $CaCO_3$ is added with the formulation, the PH value also increases. This also indicates that the formulation containing $CaCO_3$ in the base coating requires less UV radiation with higher PH value than that containing no $CaCO_3$.

Taber Abrasion

The coated (modified) Partex surface was abraded between two abradent revolving wheels applying a load of 100 g/cycle. The weight loss obtained by the sample due to such abrasion between the wheels in 100 cycles is related to the taber wear index. This means that the abrasion resistance is high, when the taber wear index is low. Results of taber wear index are shown Table II. Taking the lowest value in each case the values of taber index are shown in Figure 8 against the formulations. It is observed that abrasion resistance increases as the concentration of $CaCO_3$ increases up to 4% $CaCO_3$, and for 5% $CaCO_3$ the value of the taber index increases. The coating of formulation H (R9 + R5) shows the minimum taber index, and F (R7 + R5) or E (R6 + R5) yields the maximum taber index. This means that the coating H (R9 + R5) has the highest resistance pertained by 100 cycles in the revolving wheels compared to other coatings on the Partex surface.

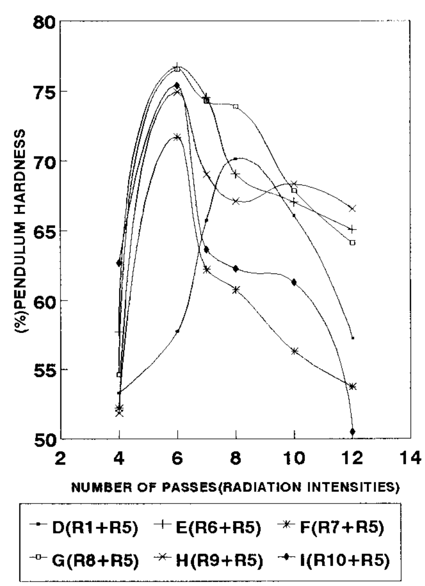

Figure 7 Pendulum hardness of UV cured polymer coatings on partex surface at different UV doses.

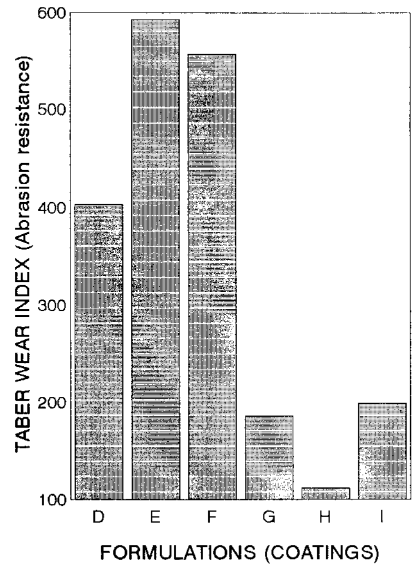

Figure 8 Taber wear index of UV cured surface coating on partex against different formulations.

Adhesion Strength

Adhesion of the UV-cured coating on the Partex surface was measured through the crosscut method, and it is observed from Table II that the coating H (R9 + R5) of formulation R9 containing 4% $CaCO_3$ with base coat has good adhesion, and there is no chipping off area during crosscut technique.

The results of adhesion force required to pull out the coating from the modified Partex surface are shown in Figure 9 against the formulations. The values of adhesion decreases as the concentration of $CaCO_3$ increases up to 3%, then it increases for 4 and 5% $CaCO_3$, but this value is still less than that given by D (R1 + R5). The adhesion values obtained by this method for all formulations containing $CaCO_3$ is somewhat less than the

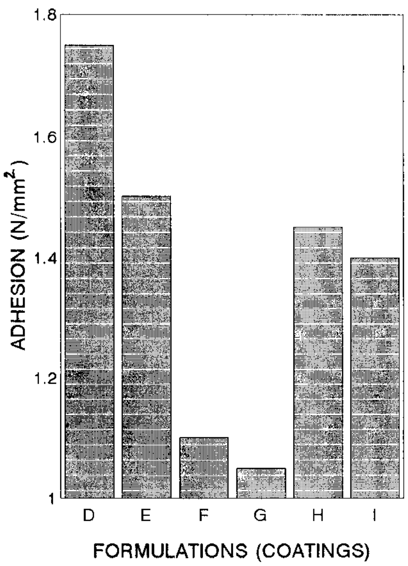

Figure 9 Adhesion force (w/mm^2) of UV cured surface coatings on partex at different formulations.

one containing no $CaCO_3$, i.e., formulation D (R1 + R5). However, the highest adhesion value is observed for formulation E (R6 + R5) containing 1% $CaCO_3$, followed by the formulation H (R9 + R5) with the 4% concentration of $CaCO_3$ in the base coat.

Weathering Effect

Samples treated with two selective formulations, namely D (R1 + R5) with no $CaCO_3$ and H (R9 + R5) containing 4% $CaCO_3$, which showed the best performance among all the formulations, were exposed to weathering treatment over a period of 500 h. The effect of weathering treatment on various properties such as PH, gloss (60°, 20°), adhesion, and MSH were determined. The results are graphically shown in Figures 10–15. In all these figures, the physical properties are plotted against weathering time or period in hours. It is observed that almost all the properties were increased initially and then began to decrease due to weathering treatment. The initial increment of the properties of the treated samples are probably caused that some unreacted radicals and other entities produced during the UV radiation using minicure were present within the treated samples and these radicals/entities were reactivated further under the UV lamps used during the weathering test.[10]

On Pendulum Hardness

The coated Partex substrates cured by UV radiation were weathered; the loss of PH is observed to be different. Although the highest PH value is found to be with sample H (R9 + R5), the loss of PH is also minimum with this sample by the weathering treatment. This is obvious from Figure 10. The maximum loss of PH by the sample H (R9 + R5) containing 4% $CaCO_3$ is 13.76%, and is

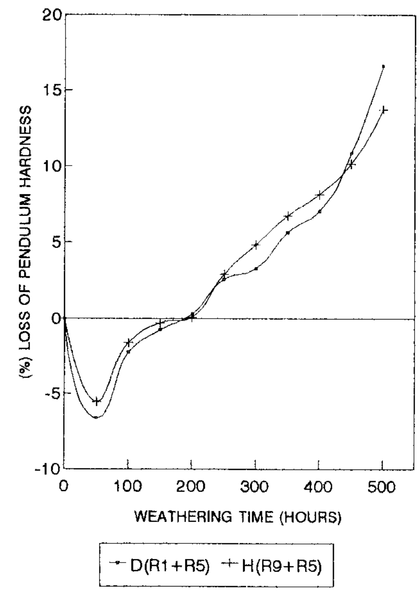

Figure 10 Effect of weathering on pendulum hardness on UV cured modified partex surface different weathering period.

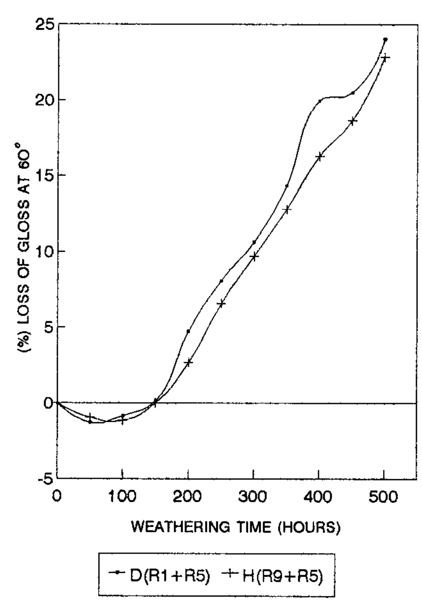

Figure 11 Effect of weathering on surface gloss at 60° on UV cured modified partex surface at different weathering period.

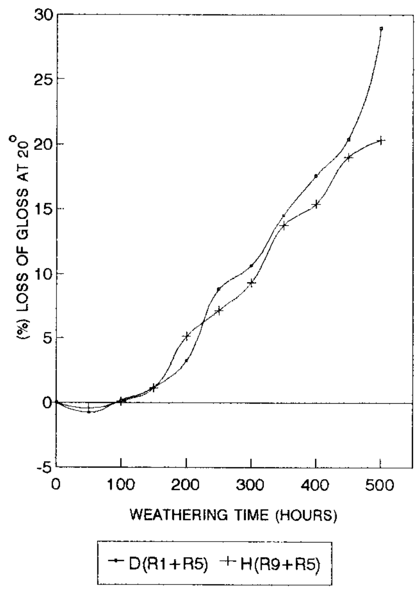

Figure 12 Weathering effect on surface gloss at 20° on UV cured modified partex surface at different weathering period.

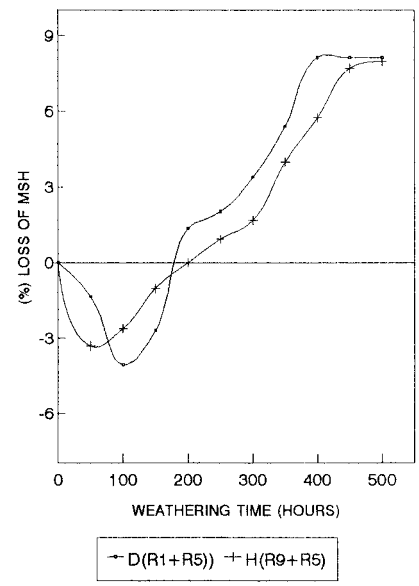

Figure 14 Effect of weathering on adhesion (w/mm^2) on UV cured surface coating period.

16.64% by the sample D (R1 + R5) having no $CaCO_3$ in the base coat.

On Gloss

The loss of gloss at 60° and 20° angles due to weathering effect are shown in Figures 11 and 12.

The highest gloss both at 60° and 20° angles before weathering treatment was observed by the sample D (R1 + R5) having no filler ($CaCO_3$), but the loss of gloss (60° and 20°) is also maximum with these sample after the weathering treatment is done. Therefore, the formulation containing 4% $CaCO_3$ have less weathering effect on its gloss

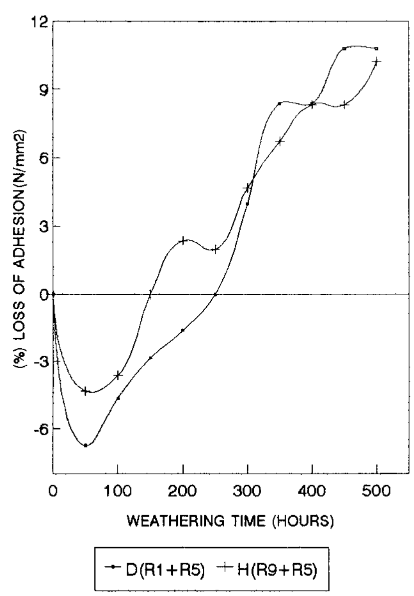

Figure 13 Effect of weathering on macro scratch hardness on UV modified partex surface at different weathering period.

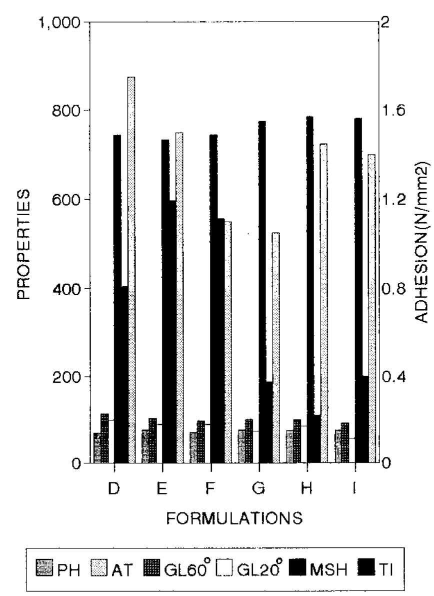

Figure 15 Various physical properties of UV cured surface coatings on partex against different formulations, only adhesion test (AT) is represented by the Y_2 axis.

properties than that of the formulation containing no filler ($CaCO_3$).

On Adhesion

The loss of adhesion strength on coated Partex is graphically shown in Figure 13. It is observed from the figure that the maximum loss of adhesion lies between 10.32 to 10.79%. The formulation H (R9 + R5) containing 4% $CaCO_3$ has less effect of weathering than that of D (R1 + R5) having no $CaCO_3$.

On Macroscratch Hardness

The effect of weathering treatment on MSH of the cured samples is shown in Figure 14. From the figure, it is observed that the loss of MSH of the formulation H (R9 + R5) 7.94% remains minimum than the formulation D (R1 + R5) 8.10%.

CONCLUSION

Various physical and mechanical properties of UV-cured polymer coatings on the Partex surface obtained from formulations R6 to R10 using top coat R5-forming coatings E (R6 + R5), F (R7 + R5), G (R7 + R5), H (R9 + R5), and I (R10 + R5) are shown in Figure 15. For better understanding, formulation D (R1 + R5) with no fillers ($CaCO_3$) is also shown. The use of fillers ($CaCO_3$) reduces the coating costs and increases the Pendulum hardness, scratch hardness, and abrasion resistance. All other properties are affected in the presence of $CaCO_3$, but the extent of decrease is not at all appreciable. The effect of weathering treatment in accelerated weathering testing also proves that the coating of the formulation H (R9 + R4) containing 4% $CaCO_3$ has less effect than that of the formulation D (R1 + R5) having no $CaCO_3$. Considering all the properties of the coatings in the whole work; it can be concluded that the coating H (R9 + R5) with the base coat R9 containing 4% $CaCO_3$ appears to be the best.

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