Comparisons of Weathering Performance of Two Automotive Refinish Coatings: A Case Study

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Received 9 April 2008; accepted 2 September 2008 DOI 10.1002/app.29341 Published online 5 December 2008 in Wiley InterScience (www.interscience.wiley.com).

ABSTRACT: The aim of this study was to compare the weathering performance of two commonly refinish coatings used in a domestic car manufacturer based on polyurethane (PU) and acid-catalyzed alkyd-melamine resins. The replacement of the alkyd-melamine refinish with the PU system was shown to largely lower the reported fading behavior of the monocoat system. For better comparison between the refinish coatings, an original equipment manufacturers' (OEM) topcoat, cured at production line, was also used. Different characterization methods including DSC, atomic force microscopy, thermo gravimetric analysis, DMTA, and Fourier transform infrared spectroscopy

were performed to elucidate the changes observed in the optical, mechanical, and weathering performance of studied coatings. It was found that the main reason for the inferior behavior of the acid-catalyzed coatings compared with that of the PU and OEM was due to its low crosslinking density. The poor weathering resistance of the former coatings was attributed to its incomplete curing reaction and therefore, to its immatured film formation. © 2008 Wiley Periodicals, Inc. J Appl Polym Sci 111: 2946–2956, 2009

Key words: refinish coatings; polyurethane; alkyd/ melamine; accelerated weathering; thermal analysis

INTRODUCTION

The general purposes of coating application in automotive industries are as follows: (1) to protect the car against environmental conditions such as humidity, sunlight, and pollutants in the air¹ and (2) to impart desirable esthetic appearance. To accomplish these functions, the coatings themselves must remain intact for a long time in a harsh environment. Photo and hydrolytic degradations are two common processes occurred, resulting in changes in chemical structure of an automotive coating.^{2–5} These chemical alterations may greatly influence the physical,⁶ mechanical,^{7–9} and electrochemical¹⁰ properties of the coating.

Therefore, protection upon weathering degradation seems to be indispensable. In recent decades, UV absorbers and hindered amine light stabilizers have been used for photo stabilization of coatings.¹¹ These usually absorb the harmful part of the solar spectrum or may deactivate free radicals to reduce photo oxidation rate.^{12,13} To lower hydrolytic degradation, several approaches such as use of coatings with highly chemical stable linkages upon water uptake have been adopted. Improved surface hydrophobic and greater crosslinking density are the two scenarios by which water permeability can be diminished.¹⁴ Air drying and refinish coatings, due to being cured at lowered temperatures, are more susceptible to permeate water leading to accelerated coating degradation.

Because of the mechanical damages (e.g., scratch and impact), which may often occur during the assembling of a car, remediation of the affected area by the aid of low temperature cured refinish coatings seems inevitable, mainly because of the heat sensitivity of the plastic parts used. From the viewpoint of weathering resistance, these coatings must be as comparable as the original equipment manufacturers' (OEM) coatings introduced in the production line. Incomparable weathering resistance of refinish coating and OEM may well lead to local defects such as gloss and/or hue variation.

Two types of refinish coatings being currently used are based on two-pack polyurethane (PU) and acid-catalyzed alkyd/melamine resins. The use of an acid catalyst is to lower the curing temperature of the OEM coatings.¹⁵ General mechanism of curing process for alkyd melamine coating in the presence of an acid catalyst is shown as a two step (A and B) in Figure 1.

The present work was aimed to attribute a reported gloss and/or hue changes occurred on different parts of a domestic car brand to the chemistries used as the refinish coating. Two different coating systems were an acid catalyzed alkyd/melamine and PU. Various analytical techniques

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Journal of Applied Polymer Science, Vol. 111, 2946–2956 (2009) © 2008 Wiley Periodicals, Inc.



Figure 1 Curing mechanism of alkyd melamine coating by acidic catalyst; reaction of melamine resin in presence of H^+ (A), reaction of alkyd resin with melamine resin (B).

including Fourier transform infrared (FTIR), Atomic force microscopy (AFM), and thermal analyses, as well as a variety of automotive testings were performed to compare the optical, mechanical, and chemical properties of the coatings against outdoor weathering and accelerated exposure.

EXPERIMENTAL

Preparation of coatings

To study the effect of material and processing parameters (primer and catalyst content), six different types of refinish and OEM automotive coatings were prepared and cured at the conditions summarized in Table I. The ratio of alkyd resin to melamine crosslinker was 70 : 30 in all of the formulations including the OEM and catalyzed ones. A derivative of an alkane sulfonic acid, provided by the coating supplier, was used as the acid catalyst to potentially lower the curing temperature. Different materials used in this work were obtained from Irankhodro Company, as shown in Table I.

The use of curing at 80°C for the catalyzed coating has two obvious reasons. The first one is to compare this sample with the PU system, which should be cured at 80°C, and the second one is because of the heat sensitivity of the plastic parts of assembled car.

CHARACTERIZATION

Hardness measurment

A Braive instrument Konig pendulum hardness tester, according to Peugeot D25 1298, was used to measure the surface hardness of different coatings. The total thickness of the coatings was between 90 and 100 μ m.

Chemical resistance

Saturated humidity test, in accordance to Peugeot D27 1571 standard, was performed to assess the resistance of the coating against humidity. MEK-rub test, according to Peugeot D27 5437 standard, was carried out to qualitatively evaluate the degree of curing.

Weathering tests

Accelerated weathering tests were performed, in accordance to the Peugeot D27 1389-95 standard, by means of an Atlas Xenotest Beta LM weather-*o*-meter containing a xenon arc light source with inner and outer quartz/quartz filters and an irradiance of 0.55 W/m^2 at 340 nm. Exposure conditions included a 102-min time interval for the dry period (relative humidity 50%, dry temperature 54°C), followed by an 18-min time interval for spraying with deionized water.

To observe the changes occurred in outdoor exposure, one set of panels were also exposed to direct natural weathering at the factory courtyard (Tehran, between July and September with min and max temperatures of 27 and 40°C, respectively, and relative humidity of 55%). Simulation of rainfall was

TABLE I Different Types of Formulations Used

Samples	Type of topcoat	Type of primer	Ratio of OEM coating to catalyst	Curing condition of topcoat $[T (°C)/t (min)]$
OEM	Alkyd/melamine	Polyester/melamine	No catalyst	140/20
CW31	Catalyzed coating	Two-pack polyester	3:1	80/20
CW41	Catalyzed coating	Two-pack polyester	4:1	80/20
CM31	Catalyzed coating	Nitrocellulose	3:1	80/20
CM41	Catalyzed coating	Nitrocellulose	4:1	80/20
PU	2-pack polyurethane	Two-pack polyester	No catalyst	80/20

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achieved daily by 20-min water spraying. The Gloss at 20° was measured by a BYK-Gardner Micro Tri-Gloss meter every week.

Surface analysis

By means of a CANADA BOMEN FTIR-ATR instrument, chemical changes of different coatings were evaluated before and after the outdoor weathering.

Surface morphology of different coatings during outdoor weathering, using a 1-cm square specimen cut before and after outdoor exposure, was studied by a DME Scanner AFM microscope DS 95-50.

Thermal analysis

Dynamic mechanical analysis was carried out on samples cured at different temperatures and contained various catalyst ratios. The temperature was increased from 20 to 145°C at a constant rate of 10°C/min with a frequency of 1 Hz. The crosslinking densities of coatings were calculated from rubbery plateau zone of storage modulus ($v_e = \frac{E}{3RT}$) where v_{er} is crosslinking density and *R* is gas constant. *E* and *T* are storage modulus and temperature at the initial point of rubbery zone, respectively.

To monitor the curing of catalyzed coatings in question, DSC studies were carried out using a Model PL DSC. Samples contained a few mg of uncured catalyzed coating. Thermographs were recorded from room temperature to 120°C at 10°C/ min. Based on the results obtained from the dynamic DSC scans, isothermal DSC regime was also performed.

For further studying of curing process of catalyzed coating, thermo gravimetric analysis (TGA) was performed from 0 to 600° C at a constant rate of 10° C/min. Previously dried formulations at room temperature, to evaporate the solvent, were used.

RESULTS AND DISCUSSION

Humidity and MEK-rub test

It was reported by the customer services at the car manufacturer that most of the cars in which color fading were shown, have been long exposed to humid environment where high chance of water permeation existed. In addition, the color alteration had been mostly seen in horizontal surfaces of the car body where sunlight may have directly shined upon. These may imply that coatings shown color fading phenomena may have not been cured properly and that crosslinking densities are not high enough. For this reason, MEK-rub as well as saturated humidity testing was performed to have an idea of how complete the curing had proceeded.



Figure 2 Gloss retention of different coatings after MEK-rub test.

MEK-rub test can be used to provide a good criterion for qualitative evaluation of network structure and solvent (swelling) resistance of coatings.¹⁶ Results of MEK-rub test for different coatings are depicted in Figure 2 in which gloss retention in percent for different coatings after the test has been measured. As shown in Figure 2, the OEM and PU coatings show the highest gloss retention among other samples. These results may clearly indicate that the catalyzed coatings, used as refinishing, have lower resistance against solvent, probably because of the lower crosslinking density. In addition, as can be seen in Figure 2, coatings with a wash-primer under layer seem to have higher solvent resistance compared with those having a mastic layer. The increase in catalyst content has not lead to a higher crosslinked density as shown for samples RW31 and RW41 as well as for RM31 and RM41. These suggest that the refinish coatings applied as the repair job are inferior to the OEM and PU. The lowered crosslinking density may result in higher water uptake when the coatings are exposed to humid environment. The effect of sunlight on coatings had also the same trend as will be discussed later in the article.

Saturated humidity test

The results of blister formation and the visual appearance of different coatings have been shown in Figure 3 and Table II. In contrary to OEM and PU coatings, which show no blistering, severe blisters are seen on the surface of different catalyzed coatings. Blistering is a result of diffusion of humidity and other soluble materials into coating.¹⁷ Therefore, the existence of blisters on the surface of the catalyzed coatings may explain the greater susceptibility of these coatings to water diffusion.

It can be also seen that in catalyzed coatings, the population of blisters are more intense in samples



Figure 3 Results of humidity test for different types of coating. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.].

primed by mastic under layer. This may be attributed to higher porosity of mastic layer, because of the existence of higher pigment loading, compared with wash primer. The results of MEK-rub and humidity testing, demonstrate that the underneath layers may be as responsible as the top refinish layer in terms of resistance against weathering exposure.

Accelerated weathering test

Three types of coatings were selected for accelerated weathering test, the results of which have been

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 TABLE II

 Results of Blistering Degrees of Different Samples

Sample type	Results of blistering
OEM	Grade 1
PU	Grade 1
RW31	Grade 3
RW41	Grade 3
RM 31	Grade 3
RM 41	Grade 4

reported in Table III. As can be seen, the catalyzed coatings show greater gloss and hue alteration during exposure, compared with OEM and PU coatings. These results are in good agreement with the results previously reported in Figures 2 and 3 and well reflect the fact that catalyzed coatings (RW31 in the Table III) show poor resistance against harsh environment. The higher gloss change of the OEM compared with that of PU may be explained because of the existence of aromatic functionalities in alkyd/ melamine formulation, making it more prone to UV absorption. The overall ranking of coatings in terms of UV resistance can therefore be shown as PU > OEM > catalyzed coating.

Outdoor weathering test

In the monocoat systems, the presence of pigment in the surface and subsequently the absorption and scattering of sun light will result in the bulk properties to remain intact. Thus, the surface properties, such as gloss and hue, can well reflect weathering performance of these coatings. Gloss variations of the samples exposed during 2 months of outdoor exposure are depicted in Figure 4. PU and OEM samples experienced least variations (almost 5%), whereas catalyzed coatings loose their gloss dramatically up to 60% during the exposure.

TABLE III Results of Accelerated Weathering Test for Different Types of Coatings

		21	0	
Type of sample	Hue changes	Gloss changes	Adhesion after test	Appearance
OEM	0.43	0.6UB	Grade e	Without any visible defect and cracks
RW31	1.35	2.1UB	Grade d	Without any visible defect and cracks
PU	0.46	0.46UB	Grade d	Without any visible defect and cracks



Figure 4 Variations of gloss versus time in simulated weathering test.

These alterations in gloss may be explained by the occurrence of microstructural changes in the surface of the coating. Therefore, to study the surface microstructure of different coatings AFM was carried out, the results of which will be shown later.

FTIR studies

By the aid of FTIR spectroscopy, chemical structure of different coatings before and after outdoor weathering test was assessed. Figure 5 shows the spectra of different coatings before and after the exposure. Although, the spectra shown in Figure 5 have not been normalized, the values given in Table IV have been obtained from the data of normalized spectra in which the intensity of C-H vibration at 2800-2900 cm⁻¹ has been taken as an internal reference and other peak vibrations have been compared with this peak. Gerlock et al.¹⁸ have shown that changes in -OH, NH absorbance area, [Δ (-OH, NH)/CH], during a constant time of exposure, can provide a criterion for comparison of weathering performance of coatings from different chemical families. These values for different coatings are summarized in Table IV. According to Table IV, the highest variations belong to catalyzed coating. For PU and OEM coatings, these values are (-0.1) and 0.76, respectively. The negative value for PU coating is somewhat surprising. It may be explained as follows: degradation of coating and completion of curing (indicated by a decrease in residual isocyanate vibration peak at 2270 cm⁻¹ and formation of urethane group at 1521 and 1218 cm⁻¹, respectively) are competing processes during exposure, especially at the beginning.⁶ At the start of weathering, because of the presence of high functional groups, the completion of curing is dominant. The decrease in (-OH,-NH) peak intensity, may be attributed to consumption of residual functional groups of PU



Figure 5 IR spectra of refinish sample before and after outdoor exposure tests.

resin during exposure. FTIR spectroscopy showed that catalyzed coating in comparison to OEM one has a lower ability to resist against environmental factors.

Hardness test

To assess the mechanical properties of different coatings, the pendulum hardness measurements were performed. Results are shown in Figure 6. The initial hardness of PU coatings and OEM ones are higher than that of those for catalyzed coatings. Because the hardness is a property that provides some approximate information regarding the extent of curing, the lower hardness of catalyzed coating may be due to partial curing of the coating at 80°C.

The results of previous different tests (MEK-rub, saturated humidity, accelerated and outdoor weathering, and hardness) are in good agreement with hardness measurements and clearly indicate that cat-

TABLE IV Chemical Changes During Outdoor Exposure for Different Coatings

		-	
	(—OH,—	NH)/CH	
Type of coating	Before outdoor exposure	After outdoor exposure	Δ [(—OH,—NH) /CH]
Catalyzed coating (RW31)	1.05	2.21	1.16
Polyurethane (PU)	2.1	2	-0.1
OEM coating	0.97	1.75	0.78

alyzed coatings have poor resistance against external environment. To obtain further clarification, the curing process of catalyzed coating was studied using thermal analysis.

Thermal analysis

Crosslinking density of thermoset coatings can be described using a DMTA analysis. Crosslinking density is a reliable criterion for assessment of curing process of coatings quantitatively. Additional investigations showed that the use of greater ratios of catalyst, that is, 3 : 1; as well as curing temperature at 80°C did not lead to refinish coatings with appropriate performance. Therefore, the effects of catalyst content and curing temperature on the performance catalyzed coating were studied. Variations of the storage modulus and loss peak (tan δ) of different samples in these studies, namely C-1 (3 : 1at 80°C), C-2 (3 : 1 at 100°C), and C-3 (2 : 1 at 100°C), are shown in Figure 7.

The crosslinking values (v_e) and the T_g of different coatings were then obtained from the plateau rubbery zone of storage modulus curve in Figure 7. T_g ,



Figure 6 Changes of hardness for different types of coating.

Journal of Applied Polymer Science DOI 10.1002/app

homogeneity of this coating, may be related to an extra amount of catalyst in the coating.

From Figure 7 it can be observed that higher ratios of acidic catalyst resulted in two loss peaks, that is, α and β . The former may be related to reaction of catalyst and coating at lower temperature. It can be argued that according to Route A in Figure 1 the introduction of acid will result in formation of $-N=CH_2$ moieties (Rout A), which are potentially more capable to accomplish the reaction of alkyd functional groups. However, excess acid will lead to formation of H $-N^+=CH_2$ moieties (Route B), which are presumably not able to fully conduct the reaction. The plausible effect would be inferior reaction of alkyd resin with melamine crosslinker in the presence of excess acid catalyst, leading to a lower crosslinking density of sample C-3 compared with C-2.

An increase in storage modulus of coating in temperature range from 120 to 140°C (near curing temperature range of an OEM coating) may be related to postcuring reaction of residual functional groups in the coating. These results may confirm an incomplete curing process of the catalyzed coatings used. The explanation of which may again confirm the inferior mechanical and optical properties previously quoted.

The low crosslinking density and incomplete curing of samples containing different concentrations of acidic catalyst may be the plausible explanation for the lowered weathering resistance of the refinish coating as a result of greater water uptake.¹⁴

Differential scanning calorimetery was also used to investigate the possibility of curing process of the coating at 80°C (conventional curing temperature of the refinish coating according to the manufacturer recommendation). Result of a nonisothermal DSC thermograph for a catalyzed coating (sample RW31) is shown in Figure 8.

It can be seen from Figure 8, that at temperature about 80°C curing has not accomplished. An exothermic peak, indicative of a curing reaction, seems to have been taken place at 106°C. The onset temperature of curing demonstrates that 80°C does not seem to be high enough for curing of this coating. The low mechanical and especially weathering

TABLE V Results of Glass Transition Temperature and Cross Linking Density

		Sample			
Parameters	OEM	C-1	C-2	C-3	
$v_e (\text{mol}/\text{cm}^3)$	0.002673	0.000486	0.000984	0.000562	
$W^{I_g}(^{\circ}C)$	85.6 85	71.1 57.1	85.1 74.5	82.7 78.2	

Figure 7 Comparison of storage modulus (up) and loss peak (down) variations of different catalyzed coatings: C-1(3 : 1 at 80°C), C-2 (3 : 1 at 100°C), and C-3 (2 : 1 at 100°C) and OEM coating.

 v_{er} and *W* (peak width in 1/2 height of tan δ) have been calculated and are listed in Table V.

Results clearly indicate that OEM coating has higher crosslinking density compared with catalyzed coatings. Lower crosslinking density of the catalyzed coatings may be related to the incomplete curing in the presence of catalyst at 80°C. However, despite the lower crosslinking density of the catalyzed coatings an increase in temperature up to 100°C (C-2) lead to higher values (in comparison to C-1). Meanwhile, the values shown in Table V may suggest that the increase in temperature and catalyst content may not necessarily result in an increase in extent of crosslinking. Lower glass transitions temperatures can be attributed to an incomplete curing process and therefore, to a lower crosslink density of coatings.

According to these observations, catalyzed coatings have lower peak width and crosslinking density than OEM coating. Also, it can be seen that higher crosslinking density result in higher peak width (lower homogeneity), except for the C-3, that in spite of its low crosslinking density, it has a higher peak width. High value of peak width, indicative of low





Figure 8 Nonisothermal DSC thermograph of the catalyzed coating (sample RW31).

performance of this coating have been influenced by this immature curing.

Subsequently, an isothermal DSC analysis was carried out at 106°C, to estimate the required time for fully curing. Result of this experiment is shown in Figure 9.

Figure 9 illustrates that curing process was terminated after a short time of almost 1.66 min (similar to the nonisothermal DSC thermograph). Termination of curing process after a short time seems to be somewhat unusual and may be explained by the loss of at least one of the required conditions for curing reaction, that is, reactants and/or catalyst. In addition, in accord to this explanation, one can observe the release of catalyst from the coating before it cures, judging from the absence of the vibration peak at 1118 cm⁻¹, corresponding to sulfonate groups in the FTIR spectra of samples in question. This can be observed from Figure 10 in which the FTIR spectra of the sample containing catalyst ratio 3 : 1 cured at 80 and 110°C, respectively. The absence of a peak appeared as a small shoulder at 1118 cm⁻¹ in the spectrum of sample cured at 110°C

may suggest that coating has somehow lost its catalyst at this temperature.

Moreover, it is clear that the exothermic peak, attributed to curing process has been followed by an endothermic peak. It may be suggested that this endothermic peak could be related to the escape of catalyst from the coating formulation. To put this observation in a test, TGA analysis was used. At first, it seems that the endothermic process can be responsible for the short curing times. After vaporization of solvent from uncured coating (at room temperature), TGA analysis was carried out on catalyzed coating, the thermograph of which is shown in Figure 11.

According to Figure 11, four weight losses can be seen, two of which are close to each other and therefore, those can be considered as a single weight loss. These are the weight losses occurred at (1) 100–170°C, (2) 250–360°C, which corresponds to one sharp DTG peak at 280°C together with a small shoulder at 340°C, and (3) 400°C, respectively. The first weight loss can be related to vaporization followed by the decomposition and oxidation



Figure 9 Isothermal thermograph of DSC analysis of catalyzed coating at 106.66°C.



Figure 10 FTIR spectra of catalyzed coating cured at 80 and 110°C.

phenomena of organics (second and third weight losses). The weight loss below 170°C could be largely attributed to the loss of volatiles from the coating formulation containing catalyst, as well as to the physically trapped solvents, and also to the release of a very small fraction of curing products (alcohol) leaving the sample pan. Therefore, it can be considered that the volatile ingredients are forced to leave at this temperature range. For this reason, an approximate calculation was made to predict and attribute the weight losses occurred below 170°C to the initial concentrations of ingredients in the formulation. Table VI reports the result of such an estimation.

It can be presumed from Table VI, that the observed weight loss exceeding 20% may largely be attributed to the release of catalyst. The plausible explanation for the rapid curing of being less than 2 min for the catalyzed coatings, based on isothermal DSC, can therefore be discussed by this phenomenon.

Atomic force microscopy analysis

Surface morphology of the coatings before and after weathering exposure was investigated by the use of an atomic force microscope (AFM). Figure 12 shows surface microstructures of different samples before and after weathering test.

Higher surface roughness of catalyzed coatings compared with that of PU and OEM coatings may be related to the incomplete curing process of catalyzed coating. The results of different roughness parameters (S_y , S_z , S_a , and S_q) are calculated and shown in Table VII. Using these parameters, R_a can be obtained. Variation of R_a before and after outdoor weathering test for different samples are shown in Figure 13.

Surface morphology after weathering clearly shows noticeable increase of surface roughness of catalyzed coatings compared with that of OEM and PU, which may be explained mostly by the chemical degradation of coating. These results are in a good agreement with the observations made in the previ-



Figure 11 TGA thermograph of a previously dried catalyzed coating (with 3 : 1 ratio).

ous experiments, in which the extent of crosslinking were shown to be higher in PU and OEM coatings. These are also consistent with the variation of gloss for different coatings weathered by artificial and outdoor exposures. It can be concluded that the PU coating outperforms the acid-catalyzed coatings when used as the refinishing job. The limitation in increasing the curing temperature of acid-catalyzed coatings in the excess of 100°C confirms the fact that PU should be introduced as the refinish instead of acid-catalyzed system, unless the type of catalyst changes to the type with a higher activity. In Figure 13 roughness of different coating is compared.

CONCLUSIONS

This study was aimed to investigate the effect of the type and the chemistry of two refinish coatings in comparison to a stoving alkyd/melamine (OEM) coating system with respect to the weathering performance. It was observed that the OEM system and the PU refinish had a higher weathering resistance compared with the acid-catalyzed one. The lower resistance of the latter system had largely affected the inferior optical and mechanical properties leading to significant hue changes. This was also systematically studied using various characterization methods. The release of the used catalyst from the coating formulation before the complete curing process resulted in a low crosslinked coating leading to poor

TABLE VI Calculation of Catalyst and OEM Content of Coating

Parameter	Catalyst part	OEM part	Total
Solvent containing coating (g)	25	75	100
Solid content	0.25	0.40	_
Solvent free coating (g)	25×0.25	75×0.4	$25 \times 0.25 + 75 \times 0.4$
Solid fraction (%)	18	82	1



Figure 12 AFM images of the coating surface before and after weathering test. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.].

TABLE VII	
Roughness of Different Coating Before and Aft	er
Weathering Test	

	Roughness parameter			
Sample type	S_y	S_z	S _a	S_q
Catalyzed-before test	129	109	7.98	11.4
Catalyzed-after test	597	487	65.5	58.2
PU-before test	72.4	57.2	4.74	6.32
PU-after test	101	82.6	4.51	7.1
OEM-before test	57.9	50	50.7	6.56
OEM-after test	68.4	63.4	6.26	8.23



Figure 13 Surface roughness of different coatings before and after weathering.

weathering resistance. On the other hand, the PU system, as an alternative refinish, outperformed the acid-catalyzed system.

The authors thank the Iran Khodro Co. for providing the coating samples.

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