

Some properties of a denture acrylic coating

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The water sorption, solubility, hardness, and color stability of thin specimens of auto-polymerized and heat-polymerized denture base polymers with and without a UV light-polymerized coating material were investigated. The specimens were saturated after storage in water for 24 h. The thin coating reduced the leakage of soluble ingredients from an auto-polymerized denture base material. The coated materials had increased surface hardness compared with the uncoated, but the difference in hardness was reduced on water uptake. A continuous increase in yellowing of the auto-polymerized coated and uncoated specimens was observed when stored in water at 37°C. □ *Color stability; glaze; hardness; surface properties; water solubility; water sorption*

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Plaque and stain accumulations are sometimes noted on polymer surfaces of fixed and removable dental prostheses. Several clinical studies have shown that denture stomatitis due to candida infection is a frequent complication for patients wearing removable dentures (1–3). Another common location for plaque retention is the surface of veneered bridge pontics, especially at the mucosal contact area, inducing inflammation in the soft tissue located toward the polymer surface (4, 5). The type and amount of a plaque layer may be dependent on the surface properties of the restorative material (6, 7).

Glaze or coating materials for composites and enamel are retained mostly by mechanical bonding (8, 9). Their intended effect on the surface properties of the composite is limited owing to reduced polymerization and wear resistance (10). However, an organic coating applied to the denture base resin may prove to be useful in preventing the undesirable deposits and, to a certain extent, degradation of the polymer surface.

The purpose of this investigation was to study the effect of a surface treatment with an organic coating material on water sorption, solubility, surface hardness, and

color stability of auto-polymerized and heat-polymerized denture base resins.

Materials and methods

The heat-polymerized (VH) and auto-polymerized (VA) denture base materials and the ultraviolet light-cured glaze material (PL) used in this investigation are listed in Table 1.

Specimen preparation and treatment

Specimen discs, 50 ± 1 mm in diameter and approximately 1.0 mm thick, were prepared in accordance with ISO 1567—that is, using a stainless steel mold mounted with gypsum in a denture flask. In accordance with the manufacturer's instructions, 1:3 and 2:7 liquid to powder ratios by weight were used for the heat- and auto-polymerized materials, respectively. The polymerization of the heat-cured resin was performed by immersion in boiling water for 20 min. The specimens were cooled at room temperature for 30 min and then left in tap water for 15 min. The polymerization of the auto-polymerized dough was performed in

Table 1. Materials used in the investigation

Material	Code	Polymerization method	Batch no.		Manufacturer
			Powder	Liquid	
Vertex RS	VH	Heat-polymerized	81052.61	83120.84	Dentimex Zeist, Holland
Vertex SC	VA	Auto-polymerized	7910292	8312082	Dentimex Zeist, Holland
Permalink GC	PL	UV-polymerized		120731	G-C Dental Industrial Corp., Japan

water at 40°C under 2.0 atm pressure for 10 min. The specimens were ground on both sides with wet carborundum papers no. 500 and no. 1000 (Struers, Copenhagen, Denmark) to a final thickness of between 0.6 and 1.1 mm. The deviation in thickness of each specimen was 0.05 mm or less over the entire surface. Eighty specimens were prepared from both types of polymers. The glaze (PL) was painted on the ground surfaces of 40 of both the heat- and the auto-polymerized specimens and cured by UV light, using a special light-curing unit (GC Perma Cure UC-1, GC Dental Industrial Corp., Tokyo, Japan) in accordance with the manufacturer's instructions. Both sides of the specimen discs were exposed for 20 min. The thickness of one layer of the coating was measured with a micrometer gauge to be $14 \pm 3 \mu\text{m}$.

Water sorption and water solubility tests

The treated and untreated series of specimens were dried in desiccators and weighed in accordance with the procedure described in the international specification for denture base polymers (11). After the constant mass of the specimens (conditioned mass) had been obtained, the discs were immersed in water at $37 \pm 1^\circ\text{C}$ for 1, 2, 4, 7, 15, 30, 45, and 60 days. After the immersion in water, the specimens were wiped dry with a hand towel and weighed. The specimens were dried in desiccators and weighed until constant mass was established. The values for water sorption and solubility were calculated as described (11).

To estimate the extent of cross-linking,

the UV-polymerized PL material was treated for several days with tetrahydrofuran (12).

Hardness tests

The resistance of organic coatings to indentation or scratching may be measured with pendulum (damping) equipment (13). Because of its long damping time and small proneness to skidding, the Albert-König pendulum (Model type 299, Erichsen GmbH and Co. Kg, Hemer-Sundwig/Westf., FRG) was used as described in DIN 53 157 (14). To read the damping time, a circular fluorescent lamp with a magnifying lens was installed close to the scale. The specimens used in the sorption and solubility tests were initially measured for hardness at three points on both sides. Each measurement was repeated twice if the standard deviation was more than 2.5% from the mean of the six measurements. After the water-saturated specimens had been weighed, the hardness test was repeated.

Color measurement

The color characteristics of the samples were measured by a fully automatic computer-controlled spectrophotometer designed for diffuse/0° reflectance. This system is based on an ultraviolet-visible light single-beam spectrophotometer (Model PMQ 3, Carl Zeiss, Oberkochen, FRG) with a reflectance attachment with gloss trap and with a viewing port 30 mm in diameter (RA3, Carl Zeiss). The spectrophotometer system was connected to a computer (Apple II, Apple Computer Inc., Cupertino, Calif.,

USA). For calibration purposes a freshly prepared white standard was used (Barium-sulfat für Weiss-standard, DIN 5033, Art. 1748, E. Merck, FRG). Colored standards (such as Hunterlab standard no. D. 33C-2263) were also used. The tristimulus values (X, Y, Z) relative to the CIE color-matching functions for CIE standard illuminant D_{65} were computed at 10-nm intervals (15).

The appearance of the specimens was characterized by means of the CIE 1976 ($L^* a^* b^*$) uniform color space (CIE LAB), which was calculated from the tristimulus values (16).

An estimated opacity of reflection efficiency was obtained by the calculation of the contrast ratio of luminous reflectance with the discs backed by a black or a white ceramic standard Y_b/Y_w (17). The black standard was a matte black-painted hollow body with the back wall covered with black velvet ($X = 0; Y = 0; Z = 0$). The procedures for specimen treatment are summarized in Fig. 1.

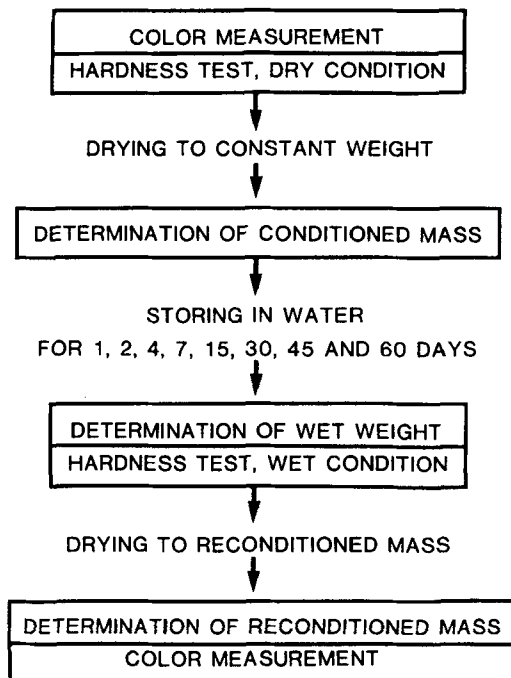


Fig. 1. A schematic presentation of the procedure used for determination of color changes, water sorption, water solubility, and hardness for the polymeric materials stored in water at 37°C.

Color stability tests

Color measurements were made before and after water sorption on the 24 heat- and the 24 auto-polymerized specimens used for water sorption and solubility. Half of the specimens were coated with the glaze.

The color stability to light and water was investigated by means of a Suntest equipment (Suntest NXE 1500, Original Hanau Quarzlampen GmbH, Hanau, FRG). One surface of each sample was continuously subjected to the radiation of the xenon light source. Color evaluations were made before weathering and after exposure for 24, 48, 96, 168, 360, and 720 h. The sample discs were covered with a 10-mm layer of distilled water. The temperature of the water was maintained at $37 \pm 5^\circ\text{C}$ by means of a thermal circulator (Thermomox 1480 THB, B. Braun, Melsungen AG, Melsungen, FRG).

Results

Water sorption and water solubility

The average of five determinations of water sorption for each period of storage in water and for each series of samples is given in Table 2. Similar results were obtained for all types of materials irrespective of storage time. Only a slight and similar increase in the quantity of solute was observed for the heat-polymerized specimens with and without glaze and for the coated auto-polymerized discs. The uncoated auto-polymerized specimens, however, showed higher values for the quantity of solute and a steeper curve for the increase in solute with time (Fig. 2).

The tetrahydrofuran treatment did not significantly affect the UV-polymerized coating material. Only a minor swelling of the polymer was observed.

Hardness test

The mean hardness values of the polymer surfaces coated and uncoated with PL are given in Table 3. The coated specimens showed a significantly higher hardness than the uncoated specimens. The hardness of all

Table 2. Water sorption of polymers ($\mu\text{g}/\text{mm}^3$) and SD

Time (h)	VH	VH + PL	VA	VA + PL
24	18.4 \pm 0.2	20.3 \pm 0.9	19.7 \pm 0.4	21.1 \pm 0.8
48	20.1 \pm 0.8	21.1 \pm 0.8	19.6 \pm 0.2	22.1 \pm 0.8
96	20.7 \pm 0.4	19.7 \pm 0.2	22.7 \pm 0.5	22.8 \pm 0.6
168	21.2 \pm 0.9	22.1 \pm 0.8	23.2 \pm 0.6	23.3 \pm 0.9
360	21.3 \pm 0.2	22.0 \pm 0.6	23.6 \pm 0.7	24.4 \pm 0.7
720	19.6 \pm 0.7	21.8 \pm 0.8	24.0 \pm 0.6	23.4 \pm 0.7
1080	21.1 \pm 0.4	21.2 \pm 0.5	20.3 \pm 1.0	23.4 \pm 0.9
1440	20.4 \pm 0.9	20.9 \pm 0.4	21.9 \pm 0.9	25.0 \pm 1.9

See Table 1 for code.

specimens decreased significantly during the first 24 h in water. Thereafter no further decrease was observed during the total observation time of 60 days. The mean hardness of wet, coated specimens was similar to that of the dry, uncoated specimens.

Color stability

Values of $L^*a^*b^*$, contrast ratio, and total color differences were calculated. Each number represents the mean of five specimens. Data obtained with a white background are shown in Fig. 3 and indicate that there is a change in color with time in water for both the coated and uncoated auto-polymerized material. Mean values of change in yellowness-blueness (b^*) versus time of

immersion in water are plotted in Fig. 4 and demonstrate that the main change in color for the auto-polymerized material is an increase in yellowness.

Tables 4–7 give color attributes of the samples before and after exposure to xenon light in water for different exposure periods. From these tables an increase in total color difference with exposure time is observed. A decrease in redness (a^*) and increase in yellowness (b^*) are also observed.

Discussion

The surface texture of an acrylic polymer is mostly determined by the processing conditions in the laboratory. A procedure to

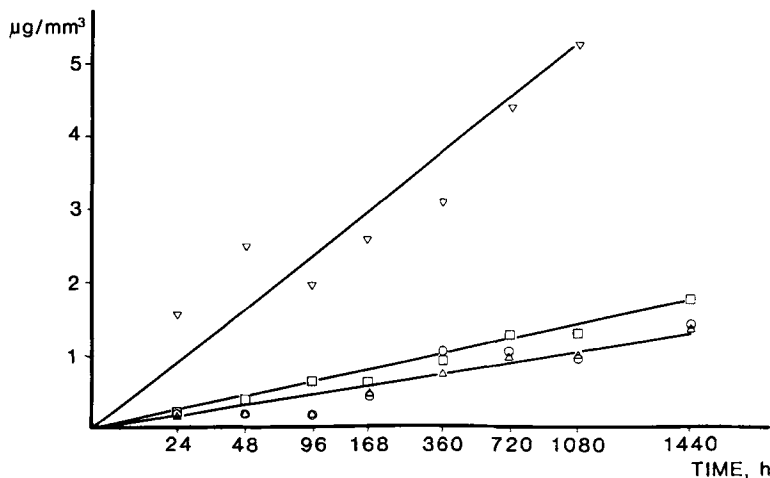


Fig. 2. The increase in the quantity of solute with time for the uncoated auto-polymerized material VA (∇), the auto-polymerized material VA coated with PL (\square), the uncoated heat-polymerized material VH (\triangle), and the heat-polymerized material VH coated with PL (\circ) when stored in water at 37°C.

Table 3. Hardness values (in s) for heat-polymerized and auto-polymerized acrylic materials carried out on dry and wet specimens

Condition	VH			VH + PL			VA			VA + PL		
	Mean	N	SD	Mean	N	SD	Mean	N	SD	Mean	N	SD
Dry	113.1	638	1.6	122.5	596	1.1	106.1	714	1.1	117.0	663	1.2
Wet	104.8	658	1.8	113.2	586	0.8	98.7	684	1.5	107.7	585	1.4

N denotes the number of measurements, and SD the standard deviation.

Fig. 3. Changes in total color difference (ΔE_{ab}^*) for specimens of an uncoated auto-polymerized material (open bars), an auto-polymerized material coated with PL (hatched bars), an uncoated heat-polymerized (closed bars), and a heat-polymerized material coated with PL (dotted bars) with increasing time when stored in water at 37°C.

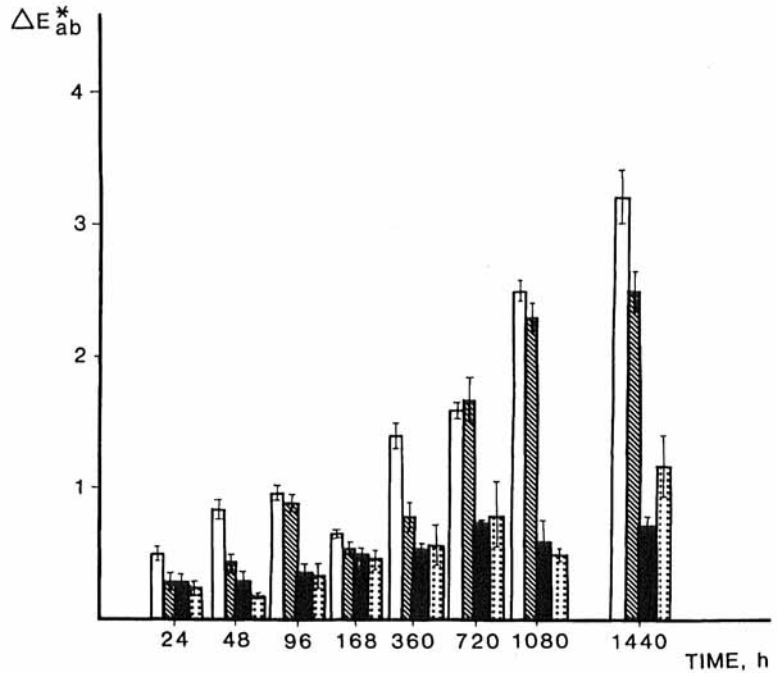


Fig. 4. Changes in yellowness-bluesness Δb^* with increasing time for specimens of an uncoated auto-polymerized material (\square), an auto-polymerized material coated with PL (∇), an uncoated heat-polymerized material (Δ), and a heat-polymerized material coated with PL (\circ) when stored in water at 37°C.

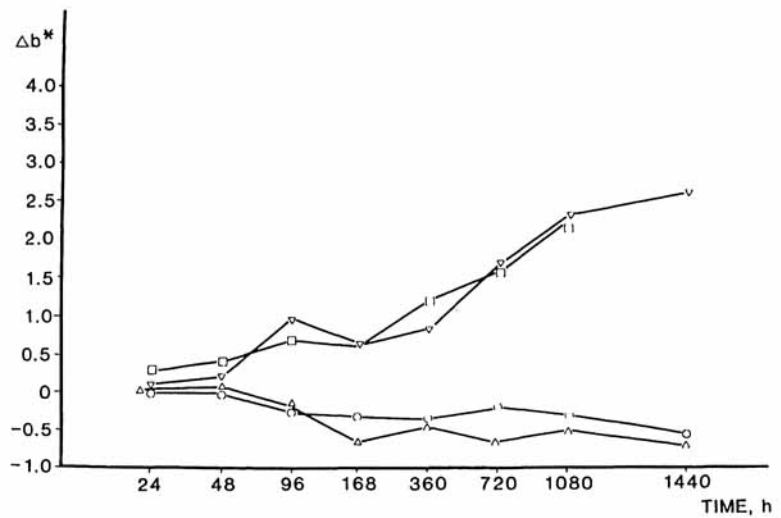


Table 4. Color attributes of specimens processed from the uncoated heat-polymerized material (VH). The measurements were made before (A) and after (B) exposure to Xe light and water at 37°C

Exposure time		L*	a*	b*	CR, %	ΔE_{ab}^*
24 h	A	81.14 ± 0.75	14.16 ± 0.96	12.41 ± 0.39	37.28 ± 1.66	1.61 ± 0.15
	B	81.38 ± 0.70	12.63 ± 0.82	11.95 ± 0.33	36.77 ± 1.44	
48 h	A	81.11 ± 0.01	13.03 ± 0.08	12.50 ± 0.14	36.24 ± 0.24	2.47 ± 0.35
	B	81.23 ± 0.15	11.57 ± 0.26	12.53 ± 0.02	35.20 ± 0.41	
96 h	A	81.67 ± 0.98	13.18 ± 1.11	11.82 ± 0.60	35.12 ± 1.93	3.37 ± 0.38
	B	81.83 ± 1.20	10.03 ± 0.74	12.99 ± 0.73	35.33 ± 1.92	
168 h	A	80.85 ± 0.77	13.84 ± 1.03	12.71 ± 0.68	35.51 ± 3.86	4.58 ± 0.02
	B	81.76 ± 0.75	9.56 ± 0.95	14.05 ± 0.48	35.85 ± 3.41	
360 h	A	80.94 ± 0.78	14.46 ± 0.96	12.49 ± 0.71	37.72 ± 1.30	7.07 ± 0.47
	B	82.15 ± 0.80	8.01 ± 0.84	15.11 ± 0.53	37.23 ± 3.16	
720 h	A	81.49 ± 0.69	13.31 ± 1.03	11.85 ± 0.61	33.42 ± 2.38	8.73 ± 0.72
	B	83.21 ± 0.83	6.71 ± 0.29	15.22 ± 0.19	36.46 ± 5.91	

improve the surface properties can be useful for patients wearing acrylic dentures or acrylic restorations. The average thickness of the present coating was smaller than a polycarbonate film coating on the fitting surface of full dentures used in a previous investigation by Engelhardt & Rehberg (18).

Relatively thin specimens were used, and thus the water uptake was independent of time of storage in water after 24 h and also independent of the type of denture base polymer. Furthermore, the use of the coating had no effect on the water uptake after 24 h; that is, it was not an effective diffusion barrier to water molecules.

Uncoated specimens from the auto-polymerized material showed a higher solubility

than the corresponding coated specimens. The coated specimens showed almost the same solubility as the coated and uncoated specimens from heat-polymerized material. This indicates that the coating of the entire specimens acts as an effective barrier for the soluble components, reducing the leakage of these components from the auto-polymerized material. This may therefore be an effective method to reduce the problem occurring when harmful or irritating constituents are leaching out of auto-polymerized materials (19).

In a clinical situation it is important that the surface of an acrylic material is smooth, since rough surfaces may favor plaque accumulation (20). To maintain a smooth

Table 5. Color attributes of specimens processed from the heat-polymerized material (VH) coated with PL. The measurements were made before (A) and after (B) exposure to Xe light and water at 37°C

Exposure time		L*	a*	b*	CR, %	ΔE_{ab}^*
24 h	A	78.30 ± 0.85	13.35 ± 1.46	13.00 ± 0.50	23.83 ± 2.02	1.03 ± 0.01
	B	78.30 ± 1.00	12.36 ± 1.51	13.20 ± 0.65	24.00 ± 3.58	
48 h	A	77.85 ± 0.01	13.95 ± 0.02	12.24 ± 0.25	23.75 ± 0.67	2.70 ± 0.04
	B	78.38 ± 0.05	11.39 ± 0.01	13.92 ± 0.21	23.65 ± 0.72	
96 h	A	78.10 ± 0.70	13.71 ± 0.53	13.40 ± 0.53	24.41 ± 1.37	3.79 ± 0.16
	B	78.58 ± 0.79	10.23 ± 0.74	14.82 ± 0.67	25.08 ± 2.35	
168 h	A	79.44 ± 0.44	11.97 ± 0.38	11.97 ± 0.15	21.01 ± 0.46	5.03 ± 0.66
	B	79.70 ± 0.43	7.17 ± 0.05	15.31 ± 0.24	21.45 ± 0.80	
360 h	A	78.92 ± 0.16	12.75 ± 0.25	12.56 ± 0.07	22.05 ± 0.21	9.39 ± 0.01
	B	78.86 ± 0.09	4.91 ± 0.11	17.72 ± 0.12	22.21 ± 0.16	
720 h	A	78.11 ± 0.35	13.65 ± 0.20	13.04 ± 0.35	22.97 ± 0.54	11.36 ± 0.18
	B	78.41 ± 0.70	3.92 ± 0.19	18.89 ± 0.00	22.77 ± 0.24	

Table 6. Color attributes of specimens processed from the uncoated auto-polymerized material (VA). The measurements were made before (A) and after (B) exposure to Xe light and water at 37°C

Exposure time		L*	a*	b*	CR, %	ΔE_{ab}^*
24 h	A	79.46 ± 1.32	13.03 ± 1.67	13.82 ± 1.15	35.86 ± 4.04	1.63 ± 0.32
	B	79.41 ± 1.21	12.70 ± 1.91	15.40 ± 0.86	37.44 ± 4.87	
48 h	A	79.98 ± 0.50	12.75 ± 0.72	12.97 ± 1.34	35.18 ± 3.80	3.38 ± 0.59
	B	79.77 ± 0.48	12.26 ± 1.85	16.22 ± 0.89	39.98 ± 9.97	
96 h	A	78.99 ± 0.11	12.88 ± 0.00	14.17 ± 0.13	34.98 ± 0.28	2.92 ± 0.23
	B	78.93 ± 0.11	11.97 ± 0.29	16.93 ± 0.02	37.96 ± 1.09	
168 h	A	79.97 ± 0.93	12.23 ± 1.64	13.40 ± 0.91	34.71 ± 3.33	3.06 ± 0.19
	B	80.05 ± 0.76	11.02 ± 1.90	16.19 ± 1.23	36.52 ± 5.90	
360 h	A	79.77 ± 0.24	12.97 ± 0.35	13.87 ± 0.22	39.09 ± 0.50	3.19 ± 0.02
	B	80.10 ± 0.05	11.16 ± 0.43	16.47 ± 0.33	44.14 ± 3.54	
720 h	A	79.40 ± 0.37	13.32 ± 0.87	14.27 ± 0.31	36.61 ± 0.43	3.19 ± 0.02
	B	80.24 ± 0.26	11.92 ± 1.07	17.81 ± 0.88	46.00 ± 1.15	

surface, the material must have a good mar resistance—that is, its ability to withstand scratching and scuffing actions, which tend to disfigure or change the surface appearance of the coating (13). Mar resistance is a surface property associated with loss of gloss. Since hardness is defined as resistance to indentation or scratching, mar resistance can be estimated by hardness testing. The measured value of hardness of an organic coating is dependent not only on the properties of the film itself but also on other factors like thickness and the nature of the coated substrate. Since the coating on the denture base polymers had an average thickness of only 14 μm , an indentation method was not suitable. The pendulum damping method of

König (14) is a sensitive method, specially designed for thin layers, and was therefore chosen for this study.

The observation that the coated specimens had higher surface hardness than the uncoated specimens is probably due to a high extent of cross-linking in the coating, as shown by the tetrahydrofuran test.

A change in appearance indicates reduction of the long-term quality of a denture. The change in total color difference and increase in yellowness when stored in water at 37°C for the coated and uncoated auto-polymerized materials is comparable to the results described by Asmussen (21) for restorative resins. This increase in yellowness (Fig. 4) is probably due to oxidation

Table 7. Color attributes of specimens processed from the auto-polymerized material (VA) coated with PL. The measurements were made before (A) and after (B) exposure to Xe light and water at 37°C

Exposure time		L*	a*	b*	CR, %	ΔE_{ab}^*
24 h	A	77.38 ± 0.67	12.00 ± 0.80	15.65 ± 0.63	24.71 ± 2.07	1.89 ± 0.31
	B	76.83 ± 0.55	10.81 ± 0.92	17.01 ± 0.34	24.24 ± 1.98	
48 h	A	77.66 ± 0.42	12.25 ± 0.91	15.28 ± 0.51	25.02 ± 2.62	3.96 ± 0.28
	B	77.11 ± 0.33	10.04 ± 0.94	18.51 ± 0.20	25.22 ± 3.00	
96 h	A	76.68 ± 0.31	12.64 ± 0.02	16.02 ± 0.33	25.99 ± 0.03	4.00 ± 1.35
	B	75.87 ± 0.02	10.77 ± 0.33	19.46 ± 0.92	26.73 ± 0.77	
168 h	A	76.89 ± 0.29	12.78 ± 0.38	16.11 ± 0.28	26.62 ± 1.35	6.58 ± 0.90
	B	75.40 ± 0.51	9.47 ± 0.24	21.59 ± 0.91	26.11 ± 1.52	
360 h	A	77.38 ± 1.01	12.13 ± 1.70	15.19 ± 1.06	24.87 ± 3.70	7.58 ± 0.69
	B	75.29 ± 0.99	7.82 ± 1.52	21.04 ± 0.28	22.73 ± 5.19	
720 h	A	77.22 ± 0.19	12.43 ± 0.17	15.58 ± 0.21	25.27 ± 0.38	6.98 ± 0.38
	B	75.91 ± 0.03	7.57 ± 0.32	20.41 ± 0.12	24.92 ± 2.93	

reactions of substances present in the auto-polymerized material.

The decrease in redness of the coated and uncoated heat-polymerized material when exposed to light and water can be explained by fading of the red pigment present in the material.

From this study it can be concluded that the coating of denture base polymers with a highly cross-linked film gives increased surface hardness. However, the hardness is decreased by water uptake, resulting in a hardness of a wet, coated material similar to that of a dry, uncoated material. A closed film can to some extent act as a barrier for dissolution of soluble ingredients from an auto-polymerized denture base material.

These observations indicate that the coating material investigated may to a certain extent increase the mar resistance of dentures. The long-term effect of the coating material in preventing undesirable deposits on dentures is, however, uncertain.

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