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Time-dependent Microhardness Gradients of Self-adhesive Resin Cements Under Dual- and Self-curing Modes

T Geng • Y Pan • Z Liu • C Yuan • P Wang • X Meng

Clinical Relevance

Acid-functional monomers in self-adhesive resin cements may decrease their self-curing polymerization ability. Light irradiation optimizes polymerization performance.

SUMMARY

Purpose: The aim of this study was to investigate Knoop microhardness of self-adhesive resin cements under dual- and self-curing modes in simulated canals for describing the polymerization behavior.

Methods and Materials: Slots in lightproof silicone cylinders with one open end were filled with the following eight materials: a traditional resin cement (Duolink), a core build-up resin material (MultiCore Flow), and six self-adhesive resin cements (RelyX Unicem 2, G-Cem Automix, Maxcem, Biscem, Multilink Speed, and PermaCem 2.0). The resins were exposed to light through the open end and then stored in a lightproof box. The Knoop hardness gradient for each resin was measured after 1 hour and 120 hours. Surface readings were obtained at 1-mm intervals from 1 mm to 10 mm away from the open ends. The data were analyzed by two-way analysis of variance and the Student-Newman-Keuls test (α=0.05).

Results: All the resin materials had stable Knoop hardness numbers (KHNs) at a certain depth; their KHNs in the self-curing mode did not change ($p>0.05$). The region above this certain depth was regarded as having undergone the dual-curing mode, and the KHN decreased gradually with depth ($p<0.05$). Between 1 and 120 hours postexposure, the ratio of the KHN at a 5-mm depth (self-cured) to that

Tengyu Geng, MDSc, The Affiliated Stomatological Hospital of Xuzhou Medical University, Xuzhou, China

Yahui Pan, MDSc, Nanjing Stomatological Hospital, Medical School of Nanjing University, Nanjing, China

Zongxiang Liu, BDS, professor, The Affiliated Stomatological Hospital of Xuzhou Medical University, Xuzhou, China

Changyong Yuan, PhD, The Affiliated Stomatological Hospital of Xuzhou Medical University, Xuzhou, China

*Penglai Wang, MDSc, professor, The Affiliated Stomatological Hospital of Xuzhou Medical University, Xuzhou, China

*Xiangfeng Meng, PhD, professor, Nanjing Stomatological Hospital, Medical School of Nanjing University, Nanjing, China

*Corresponding authors: X Meng: 30 Zhongyang Road, Nanjing, 210008, China; e-mail: mengsoar@nju.edu.cn; and P Wang: 130 Huaihai West Road, Xuzhou, 221002, China; e-mail: penglai_w@163.com

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Table 1: *Materials Used in Study*[a]

Material	Type	Composition	
		Resin Matrix	Filler
Duolink (DLK)	Conventional resin cement	Bis-GMA,TEGDMA, UDMA	Glass fillers (66 wt%)
MultiCore Flow (MCF)	Two-component core build-up material	DMA	Barium glass, ytterbium trifluoride, Ba-Al-fluorosilicate glass and highly dispersed catalysts, stabilizers and pigments (70 wt%; average, 0.04-25 μm)
RelyX Unicem 2 (RU)	Self-adhesive resin cement	TEGDMA, 2-propenoic acid, 2-methyl 1,1′-(1-[hydroxymethyl]-1,2-ethanediyl) ester dimethacrylate, 1-benzyl-5-phenyl-barbic-acid, 1,12-dodecane dimethacrylate, tert-butyl peroxy-3,5,5-trimethylhexanoate	Silanated silica, sodium persulfate, titanium dioxide, calcium hydroxide, sodium p-toluene sulfinate (70 wt%; average, 12.5 μm)
G-Cem Automix (GC)		UDMA, DMA, 4-META, phosphoric ester monomers	Fluoro-alumino-silicate glass, aluminosilicate glass fillers (65-70 wt%)
Maxcem (MC)		Bis-GMA, glycerol dimethacrylate, GPDM	Barium aluminoborosilicate glass (filler, 67 wt%; average, 3.6 μm)
Biscem (BC)		Bis-GMA, uncured DMA monomer, phosphate acidic monomer	Glass filler (filler, 60-62 wt%; average, 1.0-3.5 μm)
Multilink Speed (MS)		DMA, HEMA, acid monomers	Barium glass fillers, ytterbium trifluoride, silicon dioxide (57 wt%; average, 5 μm)
PermaCem 2.0 (PC)		Bis-GMA, UDMA, TEDMA, BPO	Barium glass (69 wt%; average, 0.02-3 μm)

Abbreviations: Bis-GMA, bis-phenol A diglycidylmethacrylate; BPO, benzoyl peroxide; DMA, dimethacrylate; GPDM; glycero-phosphate dimethacrylate; HEMA, 2-hydroxyethyl methacrylate; 4-META, 4-methacryloyloxyethyl trimellitate anhydride; TEGDMA, triethyleneglycol dimethacrylate; UDMA, urethane dimethacrylate.
[a] *Information provided by the manufacturer.*

at a 1-mm depth (dual-cured) increased in Duolink and MultiCore Flow. However, the ratios of the six adhesive resin cements varied.

Conclusion: Without light, most self-adhesive resin cements differed from traditional dual-cured resin materials in terms of Knoop microhardness, and they had a lesser capacity for chemical-induced curing.

INTRODUCTION

The first self-adhesive resin cement to be introduced and widely used was RelyX Unicem (3M ESPE, Seefeld, Germany). It was seen as a new subgroup of resin cement.[1] Self-adhesive resin cements contain acid-functional monomers that can demineralize and infiltrate the tooth substrate, which facilitates the chemical interaction between those acidic groups and hydroxyapatite and thus promotes micromechanical retention.[2,3] Therefore, self-adhesive resin cements do not require pretreatment of the tooth surface. Once the cement is mixed, its application is extremely simple.[4,5] In recent years, many new self-adhesive resin cements have been introduced, and they are widely used for all indirect restorations, such as crowns, inlays/onlays, and post cores, among others.[6-10] However, few compelling studies have been conducted to evaluate their polymerization behaviors. Adequate polymerization is still a crucial factor for obtaining optimal mechanical properties and satisfactory clinical performance of dental resin materials.

Conventional dual-cured resin materials do not contain acid-functional monomers and show a similar postcure, time-dependent increase in hardness under dual- and self-curing modes. Surface hardness increases rapidly during the first 30 minutes after exposure to light or mixing, with a subsequent slow and continuous increase until optimum hardness is achieved after 1 day.[11-14] Although the postcure increase in hardness under the self-curing mode is better than under the dual-curing mode, the final hardness that results with self-curing is always less than for the dual-cure condition. A smaller extent of slow chemical-cured compensation occurs when the intensity of light is weak.[13,14]

Compared with conventional dual-cured resin materials, self-adhesive resin cements that contain acid-functional monomers have more complex polymerization mechanisms. The dominant setting reaction of a self-adhesive resin cement involves radical-induced polymerization. Moreover, polymerization can be initiated by exposure to light or via self-curing, resulting in chemical cross-linking of monomers containing or lacking a phosphoric acid functionality.[5] Furthermore, acidic groups can react with hydroxyapatite and fillers. The acidic groups

Table 1: *Extended*

Material	Batch	Shade	Manufacturer	Recommended Light-curing Time
Duolink (DLK)	1300005927	Translucent	Bisco Inc, Schaumburg, IL, USA	≥40 s
MultiCore Flow (MCF)	S34035	Medium	Ivoclar-Vivadent, Schaan, Liechtenstein	10 s, 1100 mW/cm^2
RelyX Unicem 2 (RU)	554010	A2	3M Deutschland GmbH, Neuss, Germany	≥20 s, ≥400 mW/cm^2
G-Cem Automix (GC)	1205101	A2	GC Corporation, Tokyo, Japan	20 s
Maxcem (MC)	4619346	Clear	Kerr, Orange, CA, USA	20 s, 800 mW/cm^2; 10 s, 1100 mW/cm^2
Biscem (BC)	1400004869	Translucent	Bisco Inc, Schaumburg, IL, USA	20-30 s
Multilink Speed (MS)	S05050	Translucent	Ivoclar-Vivadent, Schaan, Liechtenstein	20 s, 800 mW/cm^2
PermaCem 2.0 (PC)	705929	Translucent	DMG; Hamburg, Germany	≥20 s, ≥400 mW/cm^2

chelate calcium in the hydroxyapatite; this results in the stable attachment of the methacrylate network to the tooth. Subsequently, ions released from the acid-soluble fillers can neutralize the remaining acidic groups to create a chelate-reinforced, three-dimensional methacrylate network.[15] Moraes and others[16] analyzed the polymerization of four self-adhesive resin cements during the initial 30-minute postcure period and found discrepancies of 11% to 79% with respect to the degree of conversion between the dual- and self-curing modes; the four self-adhesive resin cements had a slower rate of polymerization and lesser final degree of conversion than conventional resin cements under either the dual- or self-curing mode. However, it remains unclear whether the acid-base reaction initiated by the acidic monomer influences the rate of self-curing and further self-curing compensation under light of weak intensity.

In the oral environment, light shielding is inevitable, and light attenuation could be caused by the filler-volume fraction, particle size and shape, shade and thickness of composite increment, and thickness of indirect restorative material.[17-19] Therefore, the self-curing capability is vital to the successful application of dual-cured, self-adhesive resin cements. In the present study, we assessed the hardness gradient and the postcure, time-dependent change in hardness of self-adhesive resin cements in simulated root canals to determine their polymerization behaviors under dual- and self-curing modes and compared the results with those obtained with a conventional resin cement and a core build-up resin material that lacked acidic monomers. The null hypothesis was that all the dual-cured, self-adhesive resin cements would have the same postcure characteristics as the conventional resin materials.

METHODS AND MATERIALS

Test Mold and Preparation of Specimens

Eight resin materials were selected for the study: a traditional resin cement (Duolink [DLK]), a core build-up resin material (MultiCore Flow [MCF]), and six self-adhesive resin cements (RelyX Unicem 2 [RU]. G-Cem Automix [GC], Maxcem [MC], Biscem [BC], Multilink Speed [MS], and PermaCem 2.0 [PC]). Table 1 lists their compositions and recommended light-curing times.

Following the protocol used in previous studies,[13,14] the test mold was built with lightproof silicone to form a rectangular slot (width, 4 mm; height, 2 mm; length, 10 mm) with one open end. Figure 1 depicts the preparation of the test mold.

Each resin material was injected via a syringe into the silicone mold through the open end, and the excess material was squeezed out by placing a translucent microscope cover glass (0.12 mm, Matsunami Glass Inc, Osaka, Japan) over the open end. Then, the tip (Ø=7.5 mm) of a light-emitting diode

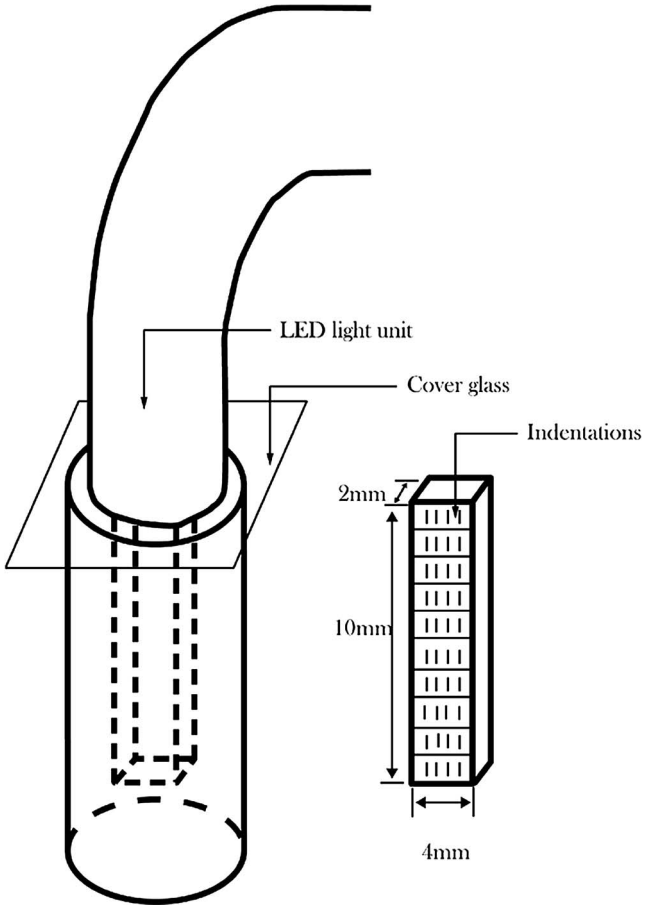

Figure 1. *Experimental setup.*

light unit (Bluephase C8, Ivoclar Vivadent, Schaan, Liechtenstein) was placed in close contact with the open end so that the opening was exposed to light intensity of 800 mW cm^{-2} for 20 seconds. This intensity was maintained with the help of a meter (Bluephase, serial number 007488, Ivoclar Vivadent). After exposure, the specimen was stored in a lightproof incubator at 37°C for 25 minutes. Then the specimen was taken from the test mold, and the test surface (4 mm × 10 mm) of each specimen was polished by thin contouring and polishing disks (Sof-Lex Extra, Lot: 70200523879; 3M ESPE) to eliminate the oxygen-inhibited layer, if any, and to achieve a polished surface for microhardness measurements.

The aforementioned procedure was carried out five times for each resin cement. Thus, five specimens were prepared and tested for each resin material per the protocol used in previous studies.[13,14,20,21]

Measurement of Knoop Microhardness

At 1 hour and 120 hours after exposure to light, the microhardness of each sample of the eight resin materials was measured at 1-mm intervals along the polished surface at distances from 1 mm to 10 mm from the open ends. The measurements were obtained using a digital microhardness tester (HV-1000, Shanghai Tester Manufactory, Shanghai, China). During each measurement, a diamond indenter in the shape of a rhombic-based pyramid was pressed into the polished surface under a load of 0.245 N for 30 seconds, and the length of the indentation's long diagonal was measured after the load was removed. The Knoop hardness number (KHN; MPa) was obtained automatically by inputting the diagonal length reading.

Ratio of Dual- and Self-curing Capability

For each resin material at 1 hour or 120 hours postexposure, the KHN at 1-mm depth (maximal dual-curing capability) was regarded as its baseline, and the KHN at 5-mm depth (self-curing capability) was compared with that of the baseline. The ratio of the 5-mm depth value to that of the 1-mm depth value was recorded for each sample.

Statistical Analysis

All data were statistically analyzed by two-way factorial design analysis of variance (dependent variable: KHN; fixed factors: postexposure time and measurement depth). The Student-Newman-Keuls test (α=0.05) was used for multiple comparisons. All statistical testing was performed by means of the software SPSS 20.0 (IBM, SPSS, Chicago, IL, USA).

RESULTS

Figure 2 presents the KHNs measured for the eight materials. The KHNs were significantly affected by each postexposure time and depth (time, $p<0.05$; depth, $p<0.05$), and a significant interaction between the two factors was identified for DLK, MCF, RU, BC, MS, and PC ($p<0.001$); there was no significant interaction for GC or MC ($p=0.443$ and $p=0.679$, respectively).

At each of the 1-hour and 120-hour postexposure time points, all the resin materials had a certain depth below which the KHN was stable. The region

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Figure 2. *Knoop hardness number gradients of eight resin materials. (A): Duolink (DLK); (B): MultiCore Flow (MCF); (C): RelyX Unicem 2 (RU); (D): G-Cem Automix (GC). (E): Maxcem (MC); (F): Biscem (BC); (G): Multilink Speed (MS); (H): PermaCem 2.0 (PC).*

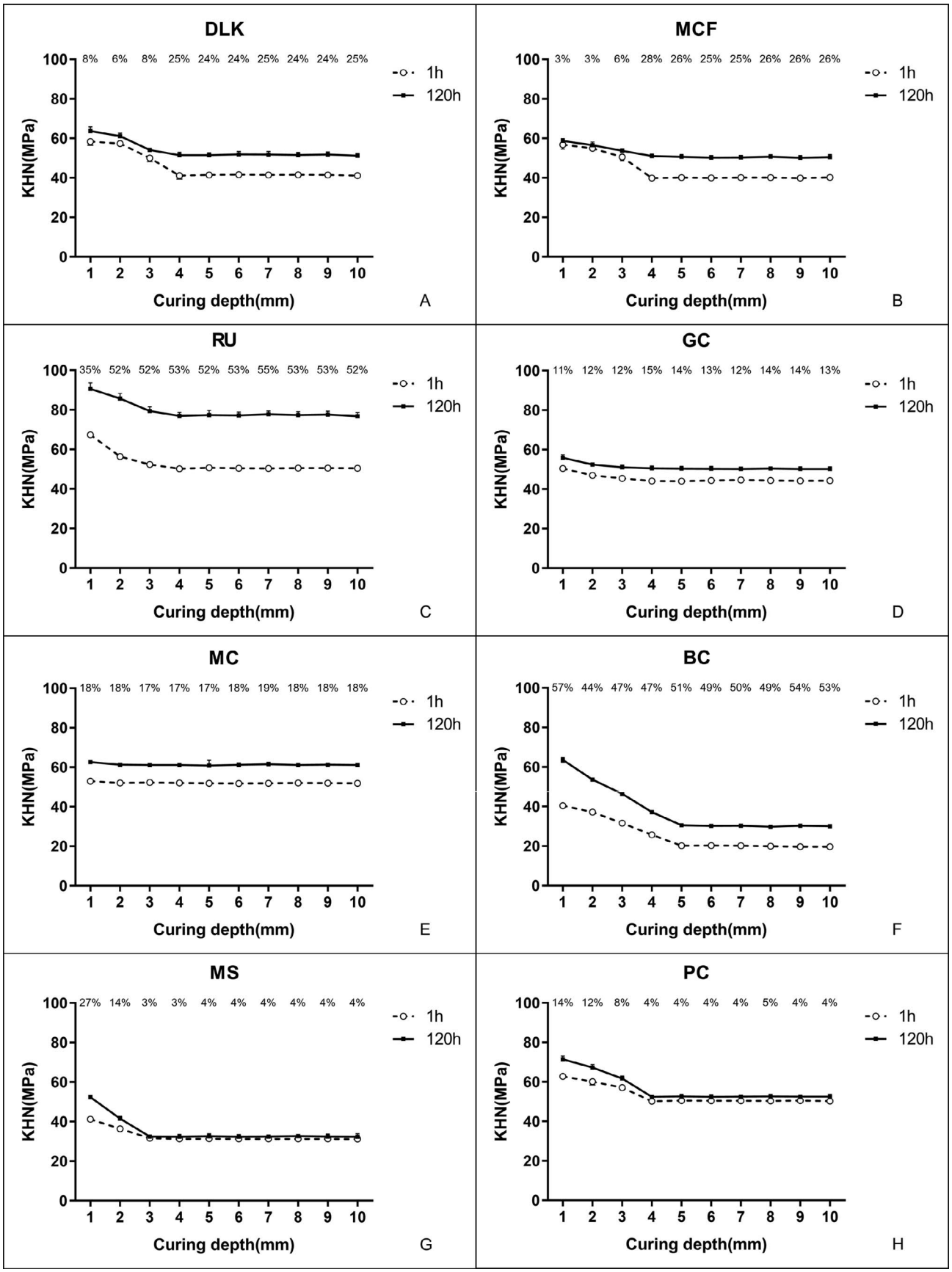

Table 2: *Ratio of Dual- and Self-curing Capability at 1 Hour and 120 Hours*[a]

Brand	1 h, %	120 h, %
DLK	70.94	81.64
MCF	70.66	86.27
RU	75.21	85.01
GC	87.38	90.05
MC	98.05	97.24
BC	49.90	47.99
MS	76.13	62.37
PC	80.43	73.63

Abbreviations: BC, Biscem; DLK, Duolink; GC, G-Cem Automix; MC, Maxcem; MCF, MultiCore Flow; MS, Multilink Speed; PC, PermaCem 2.0; RU, RelyX Unicem 2.
[a] *The ratio was obtained by dividing the Knoop hardness number at 5-mm depth (self-curing capability) by the Knoop hardness number at 1-mm depth (maximal dual-curing capability).*

above this certain depth (dual-curing area) was considered as having been through the dual-curing mode, and the KHN decreased gradually with depth through the dual-curing area ($p<0.05$). However, the material below this certain depth (self-curing area) underwent self-curing, and the KHN did not change with depth ($p>0.05$). The KHN did not change significantly at a depth >5 mm for BC; >4 mm for DLK, MCF, RU, and PC; >3 mm for GC and MS; and >2 mm for MC. Figure 2 illustrates the KHN gradients of the eight resin materials.

The KHNs of all the resin materials at 120 hours postexposure were significantly greater than those at 1 hour at all depths ($p<0.05$). The increase in KHN at 1 hour and 120 hours for each material at each distance from the light source is shown above the respective line graph (Figure 2).

Table 2 presents a comparison of the KHN ratio (5-mm depth to 1-mm depth) between the 1-hour and 120-hour postexposure time points. Between 1 hour and 120 hours after irradiation, the KHN ratio for each of the two curing modes increased from 70.94% to 81.64% for DLK (the traditional resin cement) and from 70.66% to 86.27% for MCF (the core build-up resin composite). However, the six self-adhesive resin cements had different patterns with respect to how the ratio changed over time: the ratio for RU increased from 75.21% to 85.01%, whereas the ratio for two of the materials decreased from 76.13% to 62.37% for MS and from 80.42% to 73.63% for PC. The ratio for each of the other three materials changed only marginally, that is, from 87.38% to 90.05% for GC, from 98.05% to 97.24% for MC, and from 49.90% to 47.99% for BC.

DISCUSSION

Hardness values correlate well with the degree of conversion for a specific composite.[22,23] In our present study, Knoop microhardness was selected as an index for describing the curing behavior. However, an absolute hardness value cannot be used to compare the degree of conversion among different resins owing to the potential effects of monomer, filler content, and brand.[24-26] Thus, only microhardness data for the same resin cement can be compared under different conditions.

In the present study, a conventional resin cement and a core build-up resin material that do not contain an acidic monomer had curing characteristics similar to those described previously.[13,14] In the dual-curing area, the KHN of DLK and MCF could get close to the maximum at postcure 1 hour, and after 120 hours, the increase in KHN was only 8% and 3%, respectively. In the parts of the resin that underwent self-curing, the increase in KHN (24% for DLK and 26% for MCF) was greater than that of the parts that underwent dual curing; the KHN ratio also proved this change of increase in KHN. For dual curing, the rate of light-induced polymerization was approximately 20 to 320 times faster than that of chemical polymerization, and this rapid, light-induced polymerization increases the viscosity of the composite resin.[27,28] Consequently, active radicals responsible for further self-curing become trapped.[13] Although these free radicals may come into 'contact' with the double bonds of methacrylate groups, their reactivity is limited.[13] By contrast, for self-curing, the rates of polymerization and polymer-network formation were slower. Hence, in our present study, the difference in KHN values for DLK and MCF between the postcure 1 hour and 120 hours was greater than that measured for dual curing.

The depth-dependent difference in KHN values among the six self-adhesive resin cements could distinguish the dual- and self-cure area, and the relative increase in KHN value in the self-cured areas differed between the 1-hour and 120-hour postcure samples. Various brands of self-adhesive resin cements use different types of activator/initiator systems, which might result in different mechanisms of polymerization. In general, the self-curing mode of resin materials uses benzoyl peroxide and tertiary amines as the oxidant and reductant, respectively. However, benzoyl peroxide is susceptible to radical formation, and amines can easily form salts in an acidic environment, which may greatly decrease their ability to promote polymerization.

Moreover, in the case of amines used as photoinitiators, adding sodium aryl-sulfate or aryl-borate salts can improve the acid resistance of initiators.[29,30]

RU contains sodium sulfinate salts, which may aid in preventing chemical incompatibility between acidic monomers and self-curing components. Arrais and others[31] reported that including aromatic sulfinate sodium salts in the bonding agents allowed the self-cure chemical components to initiate polymerization when light-induced polymerization was impractical. Moreover, the polymerization of RU is characterized by a rapid increase in pH, and this is probably related to the presence of calcium hydroxide in its composition. RU will achieve neutrality only 15 minutes after mixing when used in the dual-curing mode.[32] This might explain why the rate of hardening of RU in dual- and self-cure areas was similar to that of DLK and MCF, which do not contain an acid-functional monomer.

In contrast to DLK and MCF, the self-adhesive resin cements MS, PC, and BC had smaller increases in KHN values during self-curing compared with dual curing from postcure time 1 hour to 120 hours (Figure 2), and their change in KHN ratio in the dual- and self-curing modes also proved this phenomenon. It has been inferred that the acid medium may convert a conventional amine coinitiator to a protonated form, thereby deactivating the initiation system—especially when polymerization is slow (as in the case of chemical curing).[16] In our present study, the KHN ratios for the dual- and self-curing modes were 47.99% for BC and 62.37% for MS, suggesting that their polymerization under the self-curing mode was not complete, and these materials were more dependent on irradiation to achieve better mechanical properties.

The self-adhesive resin cements GC and MC had only 3-mm and 2-mm regions, respectively, in which dual curing occurred, and they had a similar increase in KHN values in the dual- and self-cure areas (Figure 2), suggesting that the curing mode has little influence on the development of hardness during the postcure period. This indicates that GC and MC might contain larger amounts of a self-cure initiator compared with the other self-adhesive resin cements that we tested. A previous study also demonstrated that GC without irradiation yielded a larger microhardness value compared with other self-adhesive resin cements,[33] whereas an amine-free redox initiator system, such as MC, might be less affected by the presence of an acid-functional monomer.[31] Although several studies have shown that use of MC results in insufficient flexural strength, elastic modulus, and bond strength compared with other self-adhesive resin cements,[31,34-36] the KHN of MC under the dual- and self-curing modes did not differ substantially from that of other self-adhesive resin cements we tested.

To date, only clinical studies of short duration have shown that the pioneering self-adhesive resin cement-RelyX Unicem could achieve acceptable adhesive effect, marginal deterioration, lower postoperative hypersensitivity, and high success rates for luting fiber and titanium posts.[8,10,37-42] However, clinical reports on other self-adhesive resin cements have been scarcely mentioned. Therefore, the selection of suitable self-adhesive resin cements should focus on their laboratory curing behavior and mechanical properties.

CONCLUSION

Clinicians should consider the effect of including an acid-functional monomer on the self-curing of self-adhesive resin cements and optimize the extent of polymerization by taking advantage of irradiation whenever possible.

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Conflict of Interest

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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Errata:

Operative Dentistry apologizes for the layout and clarity errors in the manuscripts, "Time-dependent Microhardness Gradients of Self-adhesive Resin Cements Under Dual- and Self-curing Modes", and "Effectiveness of Whitening Strips Use Compared With Supervised Dental Bleaching: A Systematic Review and Meta-analysis" published as online only articles attached to volume 45 issue 61.

Both articles were published without the final proof corrections being made. In both cases, the corrections to be made were only for style and readability and do not impact the science represented in the article.

The articles have been corrected and reposted to the website.

Our apologies to the authors and our readers for publishing content that was formatted below our standards.

The two articles affected are:

GRV da Rosa, BM Maran, VL Schmitt, AD Loguercio, A Reis, FS Naufel; Effectiveness of Whitening Strips Use Compared With Supervised Dental Bleaching: A Systematic Review and Meta-analysis. Oper Dent 1 November 2020; 45 (6): E289–E307. doi: https://doi.org/10.2341/19-160-L

T Geng, Y Pan, Z Liu, C Yuan, P Wang, X Meng; Time-dependent Microhardness Gradients of Self-adhesive Resin Cements Under Dual- and Self-curing Modes. Oper Dent 1 November 2020; 45 (6): E280–E288. doi: https://doi.org/10.2341/19-006-L