

The Effect of a Dual Cure Activator Composed of Aromatic Sulfinato Amide Derivatives on the Microhardness of Self-adhesive Resin Cements without Light Activation

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Abstract

This study evaluated the effect of a dual cure activator (DCA) on the degree of the conversion of self-adhesive resin cements (SARCs) without light activation by the mean of microhardness testing. Two hundred and seventy molds were prepared from silicone with a circular hole (5 mm diameter x 2 mm thick). The specimens were divided into the control and test groups. Each group comprised 27 specimens; three specimens for each function time of nine intervals. Five SARCs were used, and each of them was placed into the mold and covered with a mylar strip with the applied DCA. No DCA on the mylar strip served as a control. All specimens were stored in darkness in an incubator under a temperature of 37°C without light activation in each functional time. The measurements were tested by using Vickers hardness tester.

The data were statistically analyzed using two-way repeated measure ANOVA followed by Bonferroni comparison tests ($\alpha=0.05$). The overall results showed that the microhardness values in some test groups were significantly greater than the control group ($p<0.05$).

In conclusion, the addition of a DCA that represented sodium sulfonate salts increased the microhardness value of some of the SARCs.

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Introduction

At present, resin cements have a wide range of applications; such as, fixed restorations (inlay, onlay, crown and bridge, prefabricated post and orthodontic appliances due to the increasing demand for esthetics and for retention leading to a more conservative preparation design.^{1,2} The development of the demand for esthetics can be seen indirectly from various categories of ceramic materials that require tooth-like color characteristics.³ The use of resin cements requires multiple steps and is time-consuming. Moreover, newly developed resin cements that combine the easy application with the improvement of the mechanical properties and the bonded capacity in one step are self-

adhesive resin cements (SARCs).^{3,4} Recently, etching, priming and bonding have not been necessary to use in the application of SARCs. Therefore, the clinical steps have been reduced than using conventional resin cements and made simpler by eliminating the pretreatment of the dentin and the restoration surface.^{4,5} Decreasing post-operative sensitivity has also been reported as an improvement of these developed resin cements.⁶ Additionally, in some situations, a dual-cured mechanism using the combination of chemical and light activation has been needed.² Cementation of post, thick restoration and the opaque material that the light source cannot penetrate has achieved proper polymerization.^{1,2}

Though dual-cured resin cements can be cured chemically without light activation,^{1,6-9} only the rate of the polymerization is slower, but also less effective for both the physical and mechanical properties than using light.^{7,10} Furthermore, several studies have reported that chemical curing alone did not reach the maximum polymerization.^{7,11,12} Hence, both the biological and clinical properties may be affected

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if the resin cement is incompletely polymerized.⁷ As a result, early debonding of the restoration, sensitivity, marginal leakage, microleakage, and secondary caries would be a post-operative complication from the insufficient curing of the resin cements.^{8,13} Yan et al.¹⁰ reported that the polymerization was finished within 24 hours after the light activation in all tested resin cements in terms of the degree of the conversion and microhardness. In common practice, the mechanical properties would be evaluated indirectly by measuring the degree of the conversion. Microhardness is also one of the mechanical properties that provides useful information as an indirect method that can be done easily, reproducible, and saves time and costs.^{12,14-17} In addition, many studies have shown that the self-curing mechanism had the lowest degree of conversion, but few studies have reported the mechanism as time-saving.¹⁰

SARCs are an acidic functional monomer containing a luting system.⁶ Acidic functional monomers can demineralize and modify the surface of the tooth structure followed by penetrating into partially decalcified dentin and then interacting with the hydroxyapatite to create chemical bonding.^{3,18} In contrast, a residual monomer has an adverse effect, as it may affect the setting reaction of the resin cement by inactivating the tertiary amine co-initiator (TAC), especially the chemical cure mechanism because the TAC is an important activator in the chemical cure process.^{3,19} This process deactivates the free radicals and compromises the polymerization. To avoid a reaction between the acidic monomer and tertiary amine, aromatic sulfinic amide derivatives would be developed by interacting with the acidic monomer and preventing their reaction with the tertiary amine.¹⁹⁻²² This reaction would not only prevent those adverse effects, but also produce free radicals to promote free-radical polymerization.^{20,23,24} Therefore, the degree of conversion would be improved. Recently, the alternative activation system and chemical components were developed. The dual cure activator (DCA) containing sodium sulfinic salts was a developed activator to inhibit the reaction between the acidic monomer and TAC. According to Arrais et al.²³, the degree of conversion was increased when resin cement was used with the activator solution containing sodium sulfinic salts in the area where the light

was attenuated. This process not only improved the polymerization rate, but also eliminated the chemical incompatibility between the acidic monomers and the chemical curing components. However, few studies about the polymerization characteristics in terms of the chemical and dual curing modes of newly SARCs have been reported.²⁵

Because of the problem of amine inactivation and the penetration of light activation, SARCs may be affected and have a low degree of polymerization in clinical situations. Thus, the aromatic sulfinic amide derivatives are the factor that are believed to improve the polymerization. Therefore, the aim of this study was to evaluate the effect of a DCA on the degree of the conversion of SARCs without light activation by using microhardness testing.

The null hypothesis was the use of the DCA composed of the aromatic sulfinic amide derivatives with SARCs in a self-curing mode that would not affect the polymerization of the SARCs in terms of the microhardness value.

Materials and methods

Two hundred and seventy rectangular specimen molds were prepared from silicone putty (Elite HD+ putty soft Zhermack, Italy) with a circular hole at the center of the mold that measured 2 mm. in thickness and 5 mm. in diameter in accordance with ISO 6507-1:2018 (Annex A). The specimens were divided into two groups: the control and test groups.

Material	Manufacturer	Composition
RelyX™ U200 Shade: A2 Lot: 4819681	3M ESPE (St. Paul, MN, USA)	Methacrylate monomers containing the phosphoric acid groups, methacrylate monomers, silanated fillers (70 wt %/43 vol %), initiator components, stabilizers, rheological additives, alkaline(basic) initiator components, stabilizers, and pigments.
PanaviaSA luting Plus Shade: A2 Lot: 3E0173	Kuraray Medical, Inc., Tokyo, Japan	Bis-GMA, TEGDMA, HEMA, 10-MDP, hydrophobic aromatic dimethacrylate, hydrophobic aliphatic dimethacrylate, sodium fluoride, silanated barium glass filler, and silanated colloidal silica (70 wt %/40 vol %).
Maxcem Elite® Shade: yellow Lot: 7032849	Kerr, orange, CA, USA	GPDM (glycerol dimethacrylate dihydrogen phosphate), comonomers (mono-, di-, and tri-function methacrylate monomers), proprietary self-curing redox activators, photoinitiator (camphorquinone), stabilizer, barium glass fillers, fumed silica fillers, and fluoroaluminosilicate fillers.
Maxcem Elite® Chroma Shade: yellow Lot: 7145788	Kerr, orange, CA, USA	HEMA, GDM, UDMA, 1,1,3,3-tetramethylbutyl hydroperoxide TEGDMA, fluoroaluminosilicate glass, GPDM, barium glass filler, and fumed silica (69 wt %).
Multilink Speed Shade: transparent Lot: Y35409	Ivoclar Vivadent (Ellwangen, Germany)	Dimethacrylates, acidic monomers, barium glass, ytterbium trifluoride, co-polymer, silicon dioxide, initiators, stabilizers, and color pigments.
Clearfil™ DC Activator Lot: A10009	Kuraray America Inc., New York, USA	Sodium sulfonate salt and ethanol.

Table 1. Type of cements, manufacturers, lot code, and their composition of self-adhesive resin cement and dual cure activator.

The mold was placed on a glass slide with a mylar strip that was placed under the mold. Five SARC: RelyX U200; RX (3M ESPE, St. Paul, MN, USA), PanavaSA luting plus; PV (Kuraray Medical Inc., Tokyo, Japan), Maxcem Elite; ME (Kerr, orange, CA, USA), Maxcem Elite Chroma; MEC (Kerr, orange, CA, USA), and Multilink Speed; MS (Ivoclar Vivadent, Ellwangen, Germany) (Table 1) were mixed according to the manufacturer's instructions, and the circular hole was filled with mixed resin cement (Figure 1).

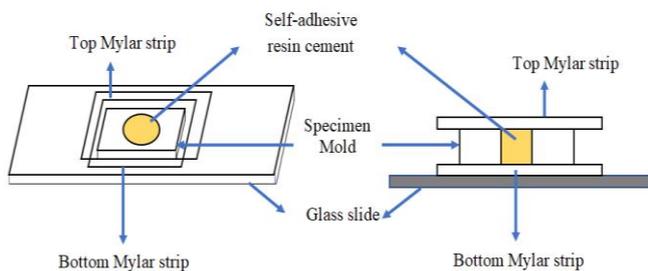


Figure 1. Resin cement sample preparation.

The ClearfilTM dual cure activator; DCA (Kuraray America Inc., New York, USA) (Table 1) was applied to another mylar strip once for each strip by dropping with a pipette to control the amount of the DCA in every sample to be equal and left to dry. The top of the mold was covered with another mylar strip as prepared above. The specimens covered with a pure mylar strip without a DCA served as a control. They were gently pressed to expel the excess resin cement, which ensured creating a smooth and flat surface to avoid an oxygen inhibiting layer. Then, they were stored in darkness in an incubator under a temperature of 37°C without light activation in a function time of nine intervals (15, 30, and 60 minutes, and 3, 6, 12, 24, 36, and 48 hours, respectively) and repeated three times for each interval ($n = 3$). The sample size was calculated by using the G power calculation program to reach more than 80% of power for the analysis.

For the microhardness evaluation, the measurement was performed in a portable darkroom at the functional time (15, 30, and 60 minutes, and 3, 6, 12, 24, 36, and 48 hours, respectively) by using the microhardness tester equipped with a Vickers indenter (Future-Tech: FM-810, Japan). The specimen samples were placed on the platform of the tester, and five indentations were applied on the top surface of each specimen after the mylar strips were

removed at 300 grams of load and 15 seconds of dwell time. The distance between the center of any indentation and the edge of the test piece was at least 2.5 times of the diameter of the indentation, and the distance between the center of two adjacent indentations was at least three times that of the diameter of the indentation. The Vickers hardness number (VHN) of each indentation was recorded and calculated as the average (mean) of these five indentations.

Statistical analysis was performed by using IBM SPSS Statistics for Windows, Version 22.0. The data were normally distributed and two-way repeated measure ANOVA was performed followed by a Bonferroni test as a post hoc examination to determine all possible pairwise comparisons. A statistical significance was considered when the p value was less than 0.05 ($\alpha = 0.05$).

Results

For the control groups, the microhardness value at 15 minutes was different in each material as shown by the descending mean values as follows: ME (23.9 VHN) > MEC (21.1 VHN) > MS (16.4 VHN) > PV (6.8 VHN) > RX (5.6 VHN). Then, the microhardness of the control groups was increased over time, and the microhardness value did not change until 48 hours. The duration of the microhardness value of the SARC displayed no change until three to 24 hours in the order of the microhardness value at the complete polymerization from the highest to the least as follows: ME > MEC > RX > PV > MS (Figure 2A). Each material showed a significant difference of the microhardness value ($p < 0.05$) by using two-way repeated measure ANOVA ($F = 3872.75$; $p = .001$).

For the test groups, the microhardness value at 15 minutes was different in each material as shown by the descending mean values as follows: ME (33.7 VHN) > MEC (30.9 VHN) > MS (25.5 VHN) > RX (7.1) > PV (6.8 VHN). After applying the DCA, the microhardness value was significantly increased when compared to the control groups that were analyzed by using two-way repeated measure ANOVA with a significance of 0.05 (95% confidence interval) except the PV that showed no further significant difference in the microhardness value. Then, the microhardness of the test groups was increased over time, and

the microhardness value did not change until 48 hours. The duration of the microhardness value of the SARC displayed no change until six to 24 hours in the order of the microhardness value at the complete polymerization from the highest to the least as follows: MEC > ME > RX > PV > MS (Figure 2B). Each material showed a significant difference of the microhardness value ($p < 0.05$) by using two-way repeated measure ANOVA ($F = 3872.75$; $p = .001$).

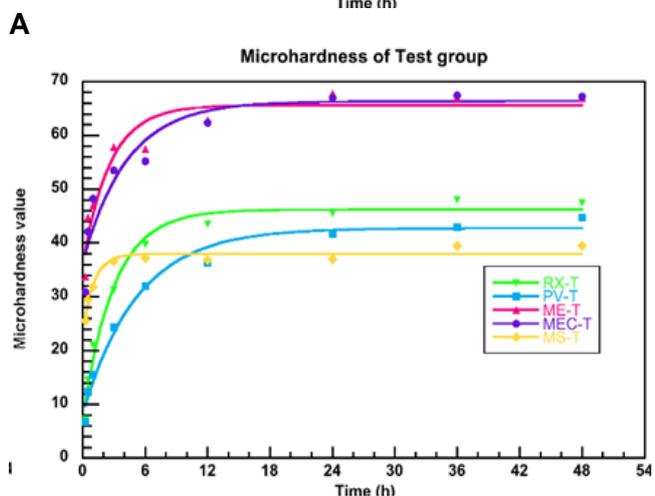
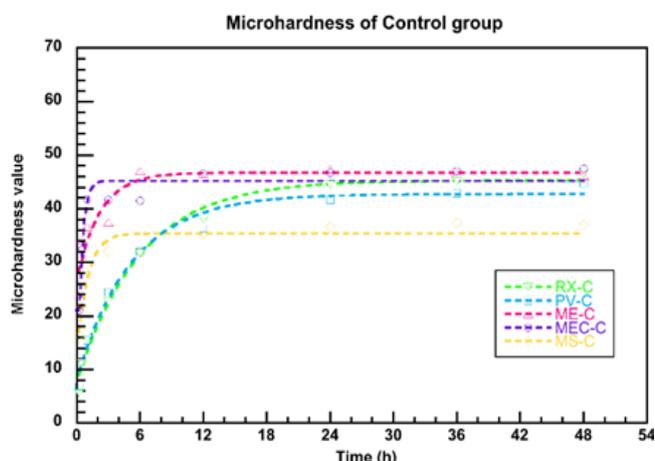


Figure 2. Microhardness value of the control (A) and test (B) groups.

The interaction among the brands, intervention and times for the VHN were clarified by additional analysis using an independent t-test that presented the microhardness value in the test groups to be significantly greater than the control group over time ($p < 0.05$). Each material had a different microhardness value at the beginning (VHN at 15 minutes) that made it difficult to compare between using the microhardness value before applying the DCA to

after applying the DCA. In order to see the effect of applying the DCA on the microhardness of the SARC more clearly, the difference between the mean microhardness value after applying the DCA (test groups) minus the mean value of the microhardness of the control group was performed (Table 2).

Time/Material	RX	PV	ME	MEC	MS
15 mins.	1.51±.66	0.97±.10	9.88±1.39	9.82±1.20	9.10±.93
30 mins.	3.28±.82	-0.99±.06	10.46±.95	9.88±1.40	7.84±.98
60 mins.	5.19±.68	1.03±.93	10.35±.1.60	6.49±.84	4.24±.67
3 hrs.	8.54±1.28	1.18±.18	20.37±1.71	11.91±1.15	4.62±.87
6 hrs.	7.99±1.03	1.51±.50	10.47±1.34	13.75±1.25	5.31±.97
12 hrs.	5.53±1.20	1.57±.37	16.27±1.38	15.70±1.39	1.58±.86
24 hrs.	0.94±.72	1.83±.80	20.28±1.59	20.25±1.05	1.43±.35
36 hrs.	2.86±1.17	1.25±.41	20.36±1.07	20.47±1.05	2.07±.75
48 hrs.	1.06±.69	2.96±.40	20.78±2.02	19.76±1.99	2.51±.87

Table 2. Mean±SD difference (Test subtracted with the Control).

Note: Mean difference is the difference between the mean microhardness value after applying the DCA (test groups) minus the mean value of the microhardness of the control group. SD: Standard deviation.

The small difference indicated applying the DCA had less effect than the microhardness. A large difference indicated applying the DCA greatly affected the microhardness value. The negative difference inferred that the microhardness value of the test groups was lower than the control group. Within the different type of materials ($F = 3872.75$; $p = .001$, tested with two-way repeated measure ANOVA), the microhardness value of the material after application with the DCA by ME showed the effect of the microhardness value had markedly increased in one and three hours. After that, the microhardness value decreased during three and six hours and increased to almost equal to three hours within 48 hours. The RX material of applying the DCA had an initial effect at 15 minutes to three hours. After that, the effect gradually decreased till 24 hours, and the applied DCA did not affect the microhardness value during 24 and 48 hours. The MEC had the microhardness value that gradually increased up to 24 hours, while MS was unaffected from 12-48 hours. On the other hand, the PV was unaffected when applied with the DCA. The ME and MEC tested groups had the highest microhardness value among all materials. The results showed that applying a DCA had the greatest effect on these two groups.

From the present result of this study, there were significant differences in the microhardness value between the test and

control groups. Therefore, the null hypothesis was rejected.

Discussion

In general, SARCs are an acidic functional monomer containing luting cements. A residual acidic monomer may affect the setting reaction of the resin cement by inactivating the TAC, especially the chemical cure mechanism because the TAC was an important activator in the chemical cure process.³ Therefore, SARCs should include another activator/initiator system; such as, aromatic sodium sulfinate salts to prevent this adverse effect.

At present, the alternative activation system has been developed. A DCA consists of a chemical co-initiator (aromatic sodium sulfinate salts, etc.) and alcohol solvent (ethanol, etc.). A study of aryl sulfinic acid sodium salts showed the sulfinic acids were highly-unstable organic chemical structures even at room temperature.²⁰ Therefore, they must be in the form of high concentration alcohol that is stable in organic compounds. The mechanism of the reaction of the aromatic sodium sulfinate salts was unclarified.²⁰ However, few research studies about the reaction of aromatic sodium sulfinate salts to the polymerization characteristics in newly SARCs have been reported. According to Kwon et al.²⁰, during the reaction of the sodium salt of aryl sulfinic acid with acidic monomer, aryl sulfinic acid and a sodium salt of the acidic monomer were produced. In the early stage, the aryl sulfinic acid (ArSO_2H) was unstable, so it was decomposed to aryl sulfonic acid (ArSO_3H) and thiolsulfonate (ArSO_2SAr). The final organic structure was an arylsulfonyl radical. Ikemura and Endo²⁴ suggested that the breakage of the aryl- SO_2H bond of the aryl sulfinic acid produced an aryl radical. The aryl radical induced the polymerization rate by helping the self-curing or redox reaction and interacting with self-etch methacrylates to prevent the tertiary amine reaction. This reaction not only prevented those adverse effects mentioned earlier, but also produced free radicals to promote free-radical polymerization.^{20,23,24}

From the overall results, the setting time of all materials was no more than 15 minutes as described from the manufacturer's instructions, and the results of the 15 minutes of both the ME

and MEC had the highest microhardness value. Therefore, the ME and MEC may be the most suitable option to repair the restoration than other materials. The microhardness value for each material increased over time. From then, the microhardness would begin to stabilize until being unchanged at 48 hours. This period, 48 hours, may be considered the optimal polymerization because the microhardness value remained unchanged. According to Baena et al.¹⁵, these studies presented the polymerization of the SARCs that tended to continue for more than 24 hours.

Based on the different results in this study, applying the DCA on to RX had an initial effect in the first 15 minutes to three hours. After that, the effect gradually decreased to 24 hours. Immediately after mixing, the RX was very acidic or had a low pH (pH~2), and the pH-value started to increase and become a neutral level (pH=7) within 24 hours. Therefore, there is a tendency when the early stages of acidity are very acidic and very active, this would allow the aromatic sodium sulfinate salts to react with the acidic monomer and cause the microhardness value to increase only in the first stage. Furthermore, when the pH would begin to adapt to the neutrality, the reaction would gradually decrease until 24 hours. As such, the value stayed constant according to the reasons mentioned above. Moreover, the manufacturer showed that the RX contain sodium sulfinate salts in their composition but according to the test group results, also found that adding DCA could increase the microhardness value since the amount of sodium sulfinate salts added to the RX may not be sufficient enough. When more was added, the sodium sulfinate salts could still react with the acidic monomer resulting in an increase in the microhardness value as opposed to the PV, which showed no further significant increase in the microhardness value compared to the control groups. The proportions of the containing aromatic sodium sulfinate salts were different for each company. There were only two materials, RX and PV, that revealed the proportion of the added sodium sulfinate salt. Characteristically, the PV could explain the proportion of containing the aromatic sodium sulfinate salts that was saturated by itself. No further acidic monomer could react with adding the DCA. Furthermore, the manufacturer claimed the activator (DCA), if used with the product from the same

manufacturer, would not be effective. In addition, the research has suggested that the pH of the PV was around 4²⁶, which was a pH value closer to the neutral than other materials. There is also another possibility to support the reason that the acidic monomer could be saturated with the activator itself. Therefore, adding DCA did not have any further effect. The polymerization rate of the PV would still be the same and the microhardness values would not increase.

While the MS remained unaffected from 12-48 hours, a few studies have examined the reaction of the MS with the activator and mentioned the MS's pH and acidity. The pH value was 4.2, which was higher than the RX, ME, MEC and PV.²⁷ The MS was self-adhesive and self-curing resin cements with a light-curing option. Several studies reported that a self-curing mechanism would provide the lowest polymerization rate,^{7,11,12} and this study showed the minimum microhardness value. The MS also had less acidity than other materials as well. One may argue that the acidic monomer, that reacts with the activator, may not be as much as other materials based on the results of this research. The microhardness value gradually increased at a slow rate and became stable at 12-48 hours.

The greatest effect was shown in the ME and MEC groups. The ME and MEC were characterized by an amine-free redox initiator system to prevent the adverse effect from the acidic monomer and improve the esthetics with the color stability.³⁰ When there was no effect from the chemical incompatibility between the acidic monomer and amine co-initiator, the microhardness value was higher than the other materials in the control groups. The MEC was different from the ME in that the MEC had a color clean-up indicator, dispensing pink before fading at the gel state, to indicate the optimal time to clean up the excess cement. The polymerization reaction of the ME and MEC showed that the microhardness values in the test groups were significantly greater than that of the control group over time and had a significantly higher microhardness value compared to other material in the test groups. The ME or MEC were probably the material that had the highest microhardness and should be applied with a DCA. The microhardness value was approximately 1.4 times higher than in the control groups and took up to 24 hours to reach the optimal polymerization. To support these results,

the previous study described that the ME and MEC had a higher component of the self-curing part.⁶ The reason for this is still unclear because of the lack of the details from the manufacturer. Another study showed the ME and MEC had an initial pH of 3.9, which increased over 24 hours to a rate of 5.5%.^{3,28,29} The acidity remained for a long time indicating that there was an acidic monomer that could react with the activator. Both reasons mentioned the slowness of the neutralization of the pH caused by the residual acidic monomer that could react with the aromatic sodium sulfinate salts. The aryl radical induced the polymerization rate by self-curing or a redox reaction. Therefore, the use of a DCA could induce the self-curing process as described above. The trend predicted that the ME and MEC had a significantly higher polymerization than other materials. Hence, the ME and MEC may be suitable in cases that the light would be unable to penetrate through the restoration. Thus, the outcome of this study correlated with Albuquerque et al.'s¹⁹ study that mentioned the importance of adding this type of salts to produce the improved polymerization of SARC's when the light source was attenuated. However, this study could only show the change in the microhardness of the SARC's after being treated with DCA but could not tell if the increasing of the microhardness affected other mechanical properties or the bond strength that must be combined in order to be useful in the clinical practice. Consequently, further investigations would be needed in order to be assembled for clinical use.

Conclusions

Within the limitations of this in vitro study, the addition of a dual cure activator that represented the sodium sulfinate salts increased the microhardness value of all the self-adhesive resin cements except Panavia SA luting plus. This finding showed the importance that adding sodium sulfinate salts improved the polymerization of the resin cements in the first minutes when the light source was attenuated.

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Declaration of Interest

The authors report no conflict of interest.

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