EGYPTIAN DENTAL JOURNAL
VOL. 65, 619:628, JANUARY, 2019
I.S.S.N 0070-9484
FIXED PROSTHODONTICS, DENTAL MATERIALS, CONSERVATIVE DENTISTRY AND ENDODONTICS
www.eda-egypt.org • Codex : 185/1901

EVALUATION OF MECHANICAL PROPERTIES OF EXPIRED AND NON-EXPIRED RESIN COMPOSITE. A COMPARATIVE STUDY

Ahmed Adel A. Aziz*

ABSTRACT

This in-vitro study evaluated the mechanical properties in terms of microhardness and degree of conversion of expired versus non-expired resin composite. A total of 120 standardized specimens were prepared for this study using Ceram.x one (E2 Enamel shade) resin composite, of which forty specimens were prepared using valid resin composite (E-0), the other forty specimens were prepared using 6 months-expired resin composite (E-6) and the last forty specimens using 12 months-expired resin composite (E-12). They were light cured using LED curing unit. 20 of each group were subjected to measurement of surface microhardness in Vickers hardness tester. The depth of cure was calculated by obtaining the microhardness ratio through dividing VHN of the bottom surface by VHN of the top surface. The other 20 specimens of each group were tested for the degree of conversion using Fourier transform infrared spectroscopy (FTIR). Data was then recorded, tabulated and statistically analyzed. The highest mean microhardness of the specimen's top was recorded for the group of 12-months expired resin composite (E-12: 83.17 ± 3.14), which was significantly different in relation to the group of 6-months expired resin composite (E-6: 72.17 ± 2.55) and the group of valid resin composite (E-0: 72.06 ± 4.20). Regarding B/T ratio (%), there were no statistical significant difference between the three groups (E-0: 69.3 ± 10.1; E-6: 66.8 ± 3.9; E-12: 67.9 ± 4.9). As for the degree of conversion results there was no statistical significant difference between the three groups (E-0: 1.48 ± 0.14; E-6: 1.68 ± 0.20; E-12: 1.74 ± 0.18). In conclusion, expired resin composite within1 year interval showed optimum and accepted mechanical properties in terms of Degree of conversion and microhardness compared to that of non-expired resin composite.

INTRODUCTION

Resin composite restorations are widely used nowadays because of their esthetic demands by the patients and also the advancements in their formulations. They are recommended for restoration of all cavity classes in anterior and posterior teeth and replaced the amalgam restorations [1,2].

Resin Composite has high mechanical properties compared to that of enamel and dentin, it is durable and have long life service. However, there are several factors which limit or affect the composite performance such as degree of conversion (DC), depth of cure and shelf life of the material [1].

* Lecturer of Conservative Dentistry, Faculty of Dentistry, Badr University in Cairo (BUC), Cairo, Egypt

The invivo and invitro performance of resin composite is affected by its mechanical composition and its degradation rate. The degradation process of this polymeric material is complex and observed as intraoral degradation and extraoral degradation due to material storage and shelf life. A lot of materials in dentistry have specific requirements for storage to maintain their properties and their shelf life [3].

The shelf life of resin composite materials is defined as the period from the manufacture date in which the composite material retains its mechanical and physical properties required to maintain its prescribed purpose. It is highly recommended by the manufacture company that the composite material should be used until reach its expiration date labelled on the material. In addition, clinically during restoration composite resin is applied by increments inside the cavity and hence only small amounts is used and as a result it reaches its expiration date before all of the material can be used [2].

The expiration date of the polymeric material is a crucial factor that should be taken into consideration, and theoretically if it has been used after its expiration date, the different properties of the material will be affected. Clinically, this may lead to a variety of consequences as discoloration, fracture, excessive wear and others [3].

Different properties whether physical and/or mechanical properties are directly influenced by the degree of conversion that has been achieved during polymerization and hence it is highly important in determining the clinical performance of such material as many properties as hardness, strength, biocompatibility and water sorption are strongly related to the degree of conversion of monomer to polymer in dental composites. Lower or insufficient degree of conversion resulted in the presence of residual unreacted monomer within the curing composite structure which acts as a plasticizer within the resin matrix and therefore resulted in a composite with inferior mechanical and physical properties with increased water absorption, poor wear resistance and greater discoloration and increase the degradation of dental composites [1,2].

Top and bottom microhardness is a property that is most frequently used to give indication about resin composite depth of cure, that is used to compare restorative materials and also used to measure the expired material deterioration. Hardness is defined as the resistance to permanent indentation or penetration. It is almost considered as a mechanical property of the dental restorations that should always be taken into consideration specially when there are large areas of masticatory forces present as in stress-bearing areas [1].

Therefore, the aim of the present study is to compare the mechanical properties in terms of microhardness and degree of conversion of expired versus non-expired resin composite.

MATERIALS AND METHODS

A commercially available esthetic composite restorative material, expired after 6 months, expired after 12 months and non-expired shelf life, Ceram.x one (E2 Enamel shade) was used for this study. Its description was shown in table 1.

A sectional Teflon mold, 2x2 (2mm in diameter and 2mm in thickness), was used to prepare the resin composite specimens. Forty standardized disc-shaped specimens of each tested material, the valid resin composite (E-0), the 6-months expired resin composite (E-6) and the 12-months expired resin composite (E-12) were prepared for this study, totaling 120 samples. This was done by applying the resin composite inside the mold cavity over glass slabs separated by transparent celluloid Mylar strip to achieve uniformly smooth surfaces and to prevent inhibition of surface polymerization then specimens were light cured for 20 seconds using Bluephase N MC LED Light Emitting Diode curing unit at zero distance to the top of the mold. The intensity of the light curing units was regularly checked using a ra-

diometer to ensure a minimum intensity of 800 mT/cm^2. The molds were placed under pressure of 1 kg from the top to remove excess material.

The resin composite syringes were stored in the refrigerator till the end of the shelf life then some resin composite were stored for extra 6 months and others were stored for 1 year.

Vickers Hardness Measurements

A total of sixty resin composite specimens have been used, twenty specimens for each group. The Vickers microhardness was determined on the top and the bottom surfaces for each specimen using a microhardness testing machine. The specimens were subjected to measurement of surface microhardness in Vickers hardness tester using a digital microhardness tester (Wilson hardness vicker's testing machine, made by Buhler, USA). To distinguish between the top and bottom of the specimens, a permanent marker was used to mark the top of each specimen. A load of 50 grams for 30 seconds was applied using a diamond microindenter. Three indentations took place for each specimen surface (top and bottom), then the Vickers hardness number VHN for each surface was calculated as the average of three readings. The Vickers hardness ratio (VHR) for each material was then calculated according to the following equation:

VHR = (bottom VHN mean value / top VHN mean value) x 100%

Data was analyzed using Statistical Package for Social Science software computer program version 22 (SPSS, Inc., Chicago, IL, USA). Data were presented in mean and standard deviation. One way Analysis of variance (ANOVA) and tukey were used for comparing data. P value less than 0.05 was considered statistically significant.

Degree of Conversion Measurements (DC%)

Degree of conversion was analyzed using Fourier transform infrared spectroscopy (FTIR). A total of sixty resin composite specimens were used for the DC % measurement, twenty specimens for each group. The resin composite samples were stored for 24 hours at room temperature and kept maintained in a dark box to be protected from light that may cause extra polymerization. Then, it was milled into fine powder using mechanical grinder and the composite powder was kept sealed in a container to avoid its exposure to light till FTIR analysis.

After that 50mg of composite powder was mixed with 5mg of potassium bromide (KBr), then this mixture was pressed to obtain thin disc which was then placed inside a specimen holder and transferred to the spectrometer for analysis.

Unpolymerized resin composite samples were obtained from each of the 3 syringes of the used resin composite and smeared to thin discs with potassium bromide then analyzed by the FTIR spectrometer and was taken as a reference for the photocured resin composite samples in relation to the degree of conversion with the same parameters as that obtained for the polymerized samples.

Recording of the absorbance peaks were obtained using the diffuse-reflection mode of the FTIR under this conditions: 32 scans, 4 cm^{-1} for the resolution over a wavelength of 300 to 400 cm^{-1}. The degree of conversion percentage of the unreacted carbon-carbon double bond (C=C %) was calculated by observing the changes occurred in the peak height ratio of the absorbance intensities of aliphatic C=C (peak at 1637 cm^{-1}) against that of an internal standard aromatic peak C=C at 1608 cm^{-1} during polymerization in relation to the uncured materials. Then the DC% was calculated for each specimen using the following equation:

$$DC\% = \left\{1 - \frac{(1637\ cm^{-1} / 1608\ cm^{-1})\ cured}{(1637\ cm^{-1} / 1608\ cm^{-1})\ uncured}\right\} x\ 100\%$$

TABLE (1) Material specifications, manufacturers and compositions

Material	Manufacturer	Descriptions/ Compositions
Ceram.x® one	Dentsply USA	•Methacrylate modified polysiloxane (organically modified ceramic) • Dimethacrylate resins • Fluorescent pigment • UV stabilizer • Stabilizer • Camphorquinone • Ethyl-4(dimethylamino)benzoate • Barium-aluminium-borosilicate glass • Silicon dioxide nano filler • Iron oxide pigments, titanium oxide pigments and aluminium sulfo silicate pigments The total amount of inorganic fillers is 72-73% wt (48-50% volume). Inorganic fillers' particle size: 0.1 to 3.0 μm.
Bluephase N MC LED Light curing unit	Ivoclar Vivadent AG, Liechtenstein, Switzerland	Light intensity = 800 mW/cm^2

TABLE (2) Variables used in this Study

Groups	Description
E-0	Group of Valid Resin Composite
E-6	Group of 6-months Expired resin composite
E-12	Group of 12-months Expired resin composite

RESULTS

Vickers Hardness Measurements

Top (T) Microhardness Results

Table 3 and Figure 1 show descriptive statistics of mean and standard deviation of top microhardness for each group.

The highest mean microhardness of the specimen's top was recorded for the group of 12-months expired resin composite (E-12: 83.17 ± 3.14), followed by the group of 6-months expired resin composite (E-6: 72.17 ± 2.55). While the lowest mean microhardness of the specimen's top was recorded for the group of valid resin composite (E-0: 72.06 ± 4.20). There was significant difference found between the group of 12-months expired resin composite (E-12) in relation to the group of 6-months expired resin composite (E-6) and the group of valid resin composite (E-0) at P-value <0.001. On the other hand, the results of both the group of 6-months expired resin composite (E-6)

TABLE (3) Comparison among different groups according to Microhardness

	E-0			E-6			E-12			P
Top	72.06	±	4.20	72.17	±	2.55	83.17	±	3.14^{ab}	<0.001*
Bottom	49.74	±	5.88	48.15	±	2.56	56.56	±	5.84^{b}	0.01*
Bottom/Top ratio (%)	69.3	±	10.1	66.8	±	3.9	67.9	±	4.9	0.79

Data expressed as mean ± SD
SD: standard deviation ***P: Probability*** ****:significance <0.05***
Test used: One way ANOVA followed by post-hoc tukey ***a: significance relative to E-0***
b: significance relative to E-6

and the group of valid resin composite (E-0) showed no statistical significant difference.

Bottom (B) Microhardness Results

Table 3 and Figure 1 show descriptive statistics of mean and standard deviation of bottom microhardness for each group.

The highest mean microhardness of the specimen's bottom was recorded for the group of 12-months expired resin composite (E-12: 56.56 ± 5.84), followed by the group of valid resin composite (E-0: 49.74 ± 5.88). While the lowest mean microhardness of the specimen's bottom was recorded for the group of 6-months expired resin composite (E-6: 48.15 ± 2.56). There was significant difference found between the group of 12-months expired resin composite (E-12) in relation to the group of 6-months expired resin composite (E-6) at P-value = 0.01. On the other hand, the results of both the group of 6-months expired resin composite (E-6) and the group of valid resin composite (E-0) showed no statistical significant difference.

B/T Ratio Results

Table 3 and Figure 2 show descriptive statistics of mean and standard deviation of B/T microhardness ratio for each group.

The highest mean microhardness of the specimen's B/T ratio (%) was recorded for the group of valid resin composite (E-0: 69.3 ± 10.1), followed by the group of 12-months expired resin composite (E-12: 67.9 ± 4.9). While the lowest mean microhardness of the specimen's B/T ratio (%) was recorded for the group of 6-months expired resin composite (E-6: 66.8 ± 3.9). There was no statistically significant difference between the three groups at P-value = 0.79.

Degree of Conversion (DC) Measurements

Table 4 & Figure 3 show descriptive statistics of degree of conversion for each group. Figure 4 shows the spectrum of the absorbance peaks using infrared spectroscopy (FTIR).

The highest mean degree of conversion was recorded for the group of 12-months expired resin composite (E-12: 1.74 ± 0.18), followed by the group of 6-months expired resin composite (E-6: 1.68 ± 0.20). While the lowest mean degree of conversion was recorded for the group of valid resin composite (E-0: 1.48 ± 0.14).

There was no significant difference between the three groups at P-value = 0.096.

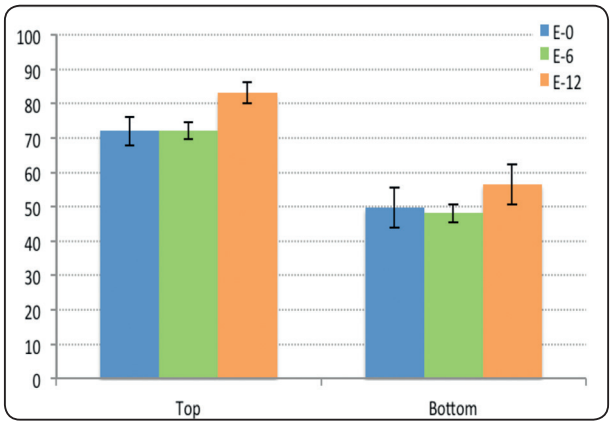

Fig. (1) representing mean of microhardness and standard deviation of the top and bottom

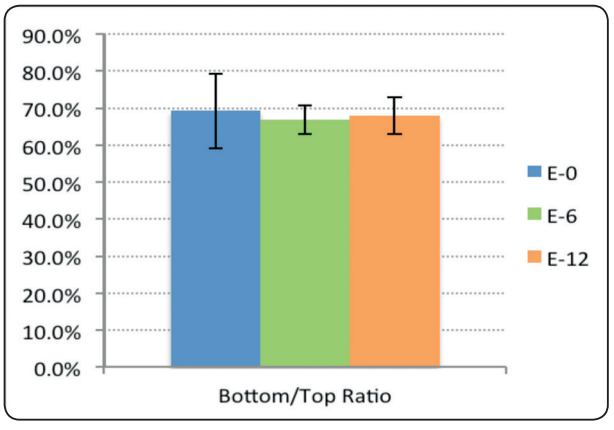

Fig. (2) representing mean of microhardness and standard deviation of the bottom/top ratio

TABLE (4) Comparison among different groups according to degree of conversion

	E-0			E-6			E-12			P
Degree of conversion (%)	1.48	±	0.14	1.68	±	0.20	1.74	±	0.18	0.096

Data expressed as mean ± SD ***SD: standard deviation*** ***P: Probability*** ****:significance <0.05***
Test used: One way ANOVA

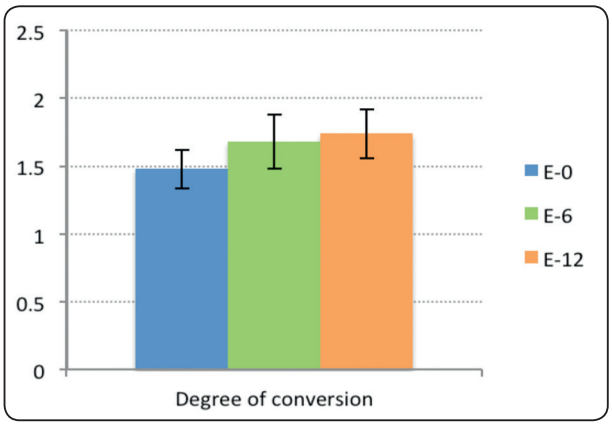

Fig. (3) Representing mean and standard deviation of the degree of conversion

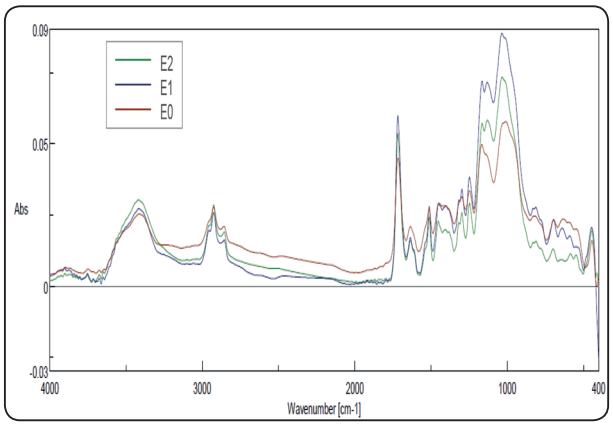

Fig. (4) Illustrating the spectrum of the absorbance peaks using FTIR of the infrared rays by C=C in the monomer of the cured composite resins of the representing three groups

DISCUSSION

This study was carried out to compare the mechanical properties in terms of microhardness (top and bottom) and degree of conversion (DC) of expired versus non-expired resin composite.

The research and development (R&D) department in the dental materials' manufacturing companies conducts many experiments, whether invitro or invivo, to study the different properties of any material before introducing it to the market. These experiments and tests were carried out for a specific period of time. Since they bear a great deal of expenses, then the shelf life of the materials are considered and labeled.

Limited researches have experimented over the effect of expiration dates on the mechanical and physical properties of esthetic dental resin composite. One of these studies has showed that there was no change in the mechanical properties of resin composite over a seven year period regardless of storage conditions [4]. Others concluded a degraded microhardness property for some of the expired dental composite materials [5, 6]. It has been found that the actual shelf life of pharmaceutical products were also exceeding their expiration dates labeled [7,8,9].

In the current study, two mechanical properties have been measured: the microhardness and the degree of conversion. The microhardness was found to be a tool that is widely used to test the curing efficiency of dental composite material [10]. The surface or top microhardness is defined as the resistance to permanent indentation or penetration and it is considered as one of the most important properties that is used to compare different restorative materials. The calculation of the bottom over top

microhardness measurements gives indirect indication about the depth of cure of composite materials that gives in turns an indication about the curing efficiency. It is applied especially to restorative materials that are used where high bite forces and stresses can exacerbate inherent material defects, resulting in inadequate fracture resistance of the materials [11,12].

Measurement of degree of conversion (DC) is a crucial tool in the determination of different composite properties whether physical, mechanical or biological properties, it was found that as the degree of conversion of composite resins increased the better the improvements of the resin composite properties [13,14].

Incomplete polymerization and lower degree of conversion affects the longevity of the resin composite restoration as this may lead to the presence of unreacted monomers that might dissolve in wet environment. Besides degradation of resin composite material occurs due to hydrolization or oxidation of the reactive site or carbon double bonds [15,16]. Moreover, the presence of uncured functional groups reduce the mechanical properties of the final restoration as it acts as plasticizers lead to reducing of the mechanical properties and is also used to compare restorative materials. On the contrary, increased polymerization results in decreasing the amount of unreacted monomers which results in a better biocompatibility restoration [17].

It is well known that factors such as light curing source, curing time, correct wavelength of curing unit and shelf life of resin composite materials can influence the degree of conversion and microhardness of resin composite material and as a result the final characteristics of composite affected. The degree of conversion is the number of carbon double bonds that are converted into single bonds and therefore resin composite obtain its optimum clinical performance. Therefore, those two properties plays an important factor in the determination of the ultimate succeed of resin composite restoration [18, 19]. The Vickers hardness is defined as the resistance to the penetration of the indentor or standing on the surface. It is considered as an important mechanical property that should be taken into consideration, especially if large areas of restoration are subjected to great masticatory forces. It is a property most commonly used to measure the expired material deterioration [12,13].

Many published researches and studies found a correlation between studying the microhardness and the degree of conversion on the opposite, others found no correlation between the degree of conversion and the microhardness among several resin composites [18,19].

In the present study, the results of mean microhardness of the specimen's B/T ratio (%) showed no significant difference between the three tested groups at P-value = 0.79. (E-0: 69.3±10.1; E-6: 66.8±3.9; E-12: 67.9±4.9). This was in agreement with Heiderscheidt et al., who found no significant difference in terms of microhardness, flexural strength and the volumetric shrinkage between the expired resin composite and non-expired ones [20]. This was also in agreement with Boroujeni et al., and Penugonda whom stated that there is no significant difference between composite past their shelf life or expiration date and unexpired resin composite [21,22].

Again, these results were in accordance with Sabbagh et al. who studied the effect of expiration date of resin composite on microhardness and found no change in the mechanical properties of resin composite in terms of microhardness between unexpired and 1-year expired resin composite resin. The microhardness results that showed non-significant difference between expired and unexpired resin composite were explained on the basis that resin composite behavior may be greatly affected by the organic part rather than inorganic part that con-

tains fillers beside coupling agent not affected [3]. On the contrary, the results was contradicted by that of Tirapelli et al. who found a significant decrease in the microhardness of expired resin composites compared to non-expired ones [6]. This difference was explained as the degradation that happened to the components of the resin composite over time especially that of its initiator which consequently resulted in incomplete cure of the polymer matrix. The results of this study also contradicted with that of Garcia et al. who evaluated the microhardness of resin composite after 180 days from their expiration date and found inferior properties in comparison to that of unexpired materials [5]. This was attributed to the increase of the remaining monomer in the expired materials and this lead to formation of polymers without typical form and hence this results in the degradation of resin composite material as stated by Ferracane [25].

Other studies found a reduction in inorganic part in terms of filler particles of resin composite when kept in liquid medium or stored for long periods and so the mechanical properties of light cured resin composites are highly dependent on the concentration of the filler particles, degree of polymerization and also the concentration of the photo initiator and consequently if this concentrations reduced the composite material will suffer degradation after its labeled shelf life [6]. Sabbagh et al., gave another explanation regarding inferior properties of expired resin composite compared to non-expired ones that there is hydrolysis through ester group linkage within the molecules or siloxane links that are formed with inorganic filler particle of the coupling agent of the expired resin composite and this results in degradation of filler surface and stress transfer generated which cause tearing away of coupling agent from filler surface that finally results in complete debonding [3]. The chemical stability of restorative materials is influenced by the degree of conversion of monmers. The presence of unreacted monmoers in there form of non-converted double carbon links act as plasticizers of resin matrix and increase the susceptibility of composite material to degradation which in turn reduces the mechanical properties.

In the present study, the results of the specimen's mean degree of conversion showed no significant difference between the three tested groups at P-value = 0.096 (E-0: 1.48 ± 0.14; E-6: 1.68 ± 0.20; E-12: 1.74 ± 0.18). These results were in agreement with Sabbagh et al. who found that there is no significant difference on some mechanical properties between expired and non-expired resin composite [3]. However, these results was contradicted by Garcia et al. [16] who evaluated the degree of conversion and other mechanical properties on resin composite past their expiration date by 180 days and found inferior properties than that of unexpired materials [5]. This was explained on the basis that there was increasing of remaining unreacted monomers in the polymeric network of the expired products which acts as a plasticizing agent and lead to material degradation and reduction of the resin composite mechanical properties [23].

Generally, the physical and mechanical properties of the polymeric materials are influenced by the percentage of monomer conversion, where increased conversion rates produces greater surface hardness, flexural strength, fracture toughness and wear resistance [24]. It was found that close to 5% of cured composite formed of residual unreacted monomers even if there is no statistical significant difference between expired and non-expired resin composite [25]. Composites that don't have elevated degree of conversion may still function well and have elevated mechanical properties due to increased concentration of inorganic filler particles and their distribution within the polymeric chain [5].

CONCLUSIONS

Under the limitations of this study, it was concluded that:

- Expired resin composite show within1 year interval optimum and accepted mechanical properties in terms of Degree of conversion and microhardness as that of non-expired resin composite.
- Further investigations have to be carried out to evaluate other physical and mechanical properties and to be done invivo in order to study the effect of the oral environmental factors as a crucial factor on these properties.
- Further researches have to be done by the manufacturing companies to investigate different properties of resin composites over more years so as to be able to increase the shelf life of resin composite if possible while retaining its properties to decrease cost of materials to doctors that will sound also in patients.

REFERENCES

1. Abed YA, Sabry HA, Alrobeigy NA. Degree of conversion and surface hardness of bulk-fill composite versus incremental-fill composite. Tanta Dent J. 2015; 12(2):71-80.
2. Eliguzeloglu Dalkilic E, Donmez N, Kazak M, Duc B, Aslantas A. Microhardness and water solubility of expired and non-expired shelf-life composites. Int J Artif Organs. 2019; 42(1):25-30.
3. Sabbagh J, Fidele Nabbout F, Edgard Jabbour E, Gaetane Leloup G. The Effect of Expiration Date on Mechanical Properties of Resin Composites. J Int Soc Prev Community Dent. 2018; 8(2):99–103.
4. Hondrum SO, Fernandez R Jr. The storage stability of dental composite resins: seven-year results. Gen Dent. 1997; 45(4):382–389.
5. Garcia Lda F, Roselino Lde M, Pires-de-Souza Fde C, Consani S. Evaluation of the conversion degree, microhardness, and surface roughness of composite resins used after their expiration date. Gen Dent. 2010; 58(6):262-267.
6. Tirapelli C, Panzeri FdC, Panzeri H, Pardini LC, Zaniquelli O. Radiopacity and microhardness changes and effect of X-ray operating voltage in resin-based materials before and after the expiration date. Mat. Res. 2004; 7(3): 409-412.
7. Lyon RC, Taylor JS, Porter DA, Prasanna HR, Hussain AS. Stability profiles of drug products extended beyond labeled expiration dates. J Pharm Sci. 2006; 95(7):1549-60.
8. American Medical Association. Pharmaceutical Expiration Dates. Report 1 of the Council on Scientific Affairs. July 25, 2001. http://www.ama-assn.org/meetings/public/annual01/csa_reports.pdf.
9. Cloud CC, Trojan TM, Suliman SN, Tantbirojn D, Versluis A: Adhesive performance of precoated brackets after expiration. Angle Orthod. 2016; 86(2):235-240.
10. Mousavinasab SM, Meyers I. Comparison of Depth of Cure, Hardness and Heat Generation of LED and High Intensity QTH Light Sources. Eur J Dent. 2011; 5(3): 299-304.
11. Mills RW, Uhl A, Blackwell GB, Jandt KD. High power light emitting diode (LED) arrays versus halogen light polymerization of oral biomaterials: Barcol hardness, compressive strength and radiometric properties. Biomaterials. 2002; 23(14):2955-2963.
12. Sobrinho LC, Goes MF, Consani S, Sinhoreti MA, Knowles JC. Correlation between light intensity and exposure time on the hardness of composite resin. J Mater Sci Mater Med. 2000; 11(6):361-364.
13. Galvão MR, Caldas SG, Bagnato VS, de Souza Rastelli AN, de Andrade MF. Evaluation of degree of conversion and hardness of dental composites photo-activated with different light guide tips. Eur J Dent. 2013; 7(1):86-93.
14. Lovell LG, Lu H, Elliott JE, Stansbury JW, Bowman CN. The effect of cure rate on the mechanical properties of dental resins. Dent Mater. 2001; 17(6):504-511.
15. Oysaed H, Ruyter IE, Sjøvik Kleven IJ. Release of formaldehyde from dental composites. J Dent Res. 1988; 67(10):1289-1294.
16. Yap AU, Wong NY, Siow KS. Composite cure and shrinkage associated with high intensity curing light. Oper Dent. 2003; 28(4):357-364.
17. Asmussen E, Peutzfeldt A. Influence of pulse-delay curing on softening of polymer structures. J Dent Res. 2001; 80(6):1570-1573.

18. Witzel MF, Calheiros FC, Gonçalves F, Kawano Y, Braga RR. Influence of photoactivation method on conversion, mechanical properties, degradation in ethanol and contraction stress of resin-based materials. J Dent. 2005; 33(9):773-779.

19. Cekic-Nagas I, Egilmez F, Ergun G. The effect of irradiation distance on microhardness of resin composites cured with different light curing units. Eur J Dent. 2010; 4(4):440-446.

20. Heiderscheidt EC, Lise DP, Nunes RAC, Baratieri LN. Microhardness, flexural strength and volumetric shrinkage of composites after expiration date. Dent Mater. 2011; 16(3):81-84.

21. Boroujeni PM, Mohamadi S. Microhardness of two types Composites Before and After Expiration Date. IADR 2007, Abstract (Kuwaiti Division).

22. Penugonda B. Evaluation of Expired Composite Materials Efficacy, New York University, USA, Brisbane IADR 2006, Abstract #2127.

23. Spyropoulou N, Plevritaki A, Masouras K, Lagouvardos P. Color Stability of Expired Restorative Dental Composite Resins. IOSR-JDMS 2017; 16(3):80-85.

24. Lovell LG, Berchtold KA, Elliott JE, Lu H, Bowman CN. Understanding the kinetics and network formation of dimethacrylate dental resins. Polym Adv Technol 2001; 12: 335-345.

25. Ferracane JL. Elution of leachable components from composites. J Oral Rehabil 1994; 21(4): 441-452.