

<p><b>Research Report</b></p>
-------------------------------

## The effect of self-etch and total-etch bonding systems application on microleakage of bulkfill flowable composite restoration in carbonated drink immersion

Widya Saraswati, Aurny Thania Song Hadinata and Sukaton

Department of Conservative Dentistry,  
Faculty of Dental Medicine, Universitas Airlangga,  
Surabaya, Indonesia

### ABSTRACT

**Background:** Resin composite is more advanced today, but the marginal leakage still cannot be avoided, for it takes the bonding system. Bonding system aims to provide a layer between a substrate and composite so it has a strong bond. The current latest bonding system is called universal bonding. It can be used with 2 techniques, both self-etch and total-etch. Universal bonding contains 10MDP so there's no need for etching and priming. 10MDP results in better adhesion than previous bonding monomers generation. A total-etch technique require a pre-etching step to remove smear layer so that it formed porous as a retentive sites. **Purpose:** Distinguish the marginal leakage of flowable bulkfill composite overlays with the application of total-etch and self-etch bonding systems accompanied by immersion of carbonated beverages. **Methods:** Tooth samples were prepared to a depth of 2mm, then the bonding agent was applied directly to the cavity without etching (self-etch), etched before the application of bonding agent (total-etch), directly loaded without pre-etching and bonding (control). using a flowable bulkfill composite as a material restorative. All of the sample both total-etch and self-etch soaked in Cola drinks for 5 minutes then repeated until 10 treatment each day (one day is assumed to be 12 hours) up to 7 days, followed by immersion in the methylene blue 0.5 % for 24 hours. Sample then splitted buccolingual direction, leakage was assessed by scoring the edge trough the depth of methylene blue penetration. **Results:** Mann-Whitney test showed a significance differences ( $P > 0.05$ ) each group. **Conclusion:** Marginal leakage of composite restoration with total-etch technique and self-etch technique is smaller than the control group, and the marginal leakage of composite restoration with total-etch technique is smaller than that of self-etch group.

**Keywords:** self-etch; total-etch; universal bonding; flowable bulkfill; edge leakage; carbonated drinks

Correspondence: Widya Saraswati, Department of Conservative Dentistry, Faculty of Dental Medicine, Universitas Airlangga, Jl. Prof. Moestopo No. 47, Surabaya. E-mail: widya-s@fkg.unair.ac.id

### INTRODUCTION

Composite resin is one of the tooth-colored material that is widely used today because it has a higher aesthetic value than other material. Composite resins harden through the polymerization process. This polymerization will make composite resin susceptible to marginal leakage.<sup>1</sup> Another factor that affects marginal leakage between teeth and restorations in addition to shrinkage due to polymerization process and patient dietary habits.<sup>1</sup>

Currently, soft drinks consumption has increased dramatically. Soft drinks contain simple carbohydrates in high concentrations such as glucose, fructose, sucrose and other simple sugar contents. Generally soft drinks contain carbonic acid, phosphoric acid, malic acid, citric acid, tartaric acid with a low pH.<sup>2</sup> According to the researchers, carbohydrate drinks damage the surface of the teeth, which

are caused by sugar or other factors such as the acidity pH contained therein.<sup>3</sup> A tooth will demineralize and demineralize, when the salivary pH falls below 5.5, the demineralization process will be faster than remineralization.<sup>3,4,5</sup> Many efforts have been made to prevent marginal leakage between tooth surfaces and composite resins, including the use of effective adhesion agents and new types of composites to reduce microleakage.<sup>2,6,7,8</sup>

There's a new composite matrix that has been developed recently; it's called bulkfill composite resin. Bulkfill composite resin does not require of *multi-layer* application. Bulk-fill composites are claimed to be restorative materials used in deep preparations and effectively photoactivated in layers up to 4 mm. It can minimize polymerization shrinkage.<sup>2,6,7</sup> Bulkfill composite resins have less polymerization shrinkage than conventional composites. This is due to the addition of a special *modifier* that can reduce the stress

*shrinkage* due to the *curing* process. Composite *bulkfill* itself consists of 2 types, named *flowable bulkfill* which has low viscosity and *packable* which has high viscosity. This is intended to improve marginal adaptation so as to reduce edge leakage.<sup>6</sup>

There is a discovery of a new bonding system that is currently classified into two systems, namely: *total-etch* (*etch and rinse*) and *self-etch* (without flushing). The *self-etch* primers can perform etching and *priming* functions simultaneously, so the clinical procedure is also simpler than the *total-etch*.<sup>1,7,8</sup> Bonding *total-etch* has a long procedure so that it makes the dentin dry out easily and results in collagen fiber collapsing.<sup>8,9</sup> In the *self-etch* system, there is no etching procedure in advance which causes the pH of the *self-etch* bonding system to be not too acidic so that *self-etch* does not cause dentin demineralization, but *self-etch* also has a disadvantage, namely not strong attachment to enamel.<sup>10</sup>

According to research conducted by Burgess *et al*<sup>11</sup> for 2 years, there was no significant difference in clinical performance between *total-etch* and *self-etch* during these 2 years. *Self-etch* shows good retention strength for 2 years. According to Bolgul *et al* use of *self-etch 2 bottle* (6th generation bonding) and restoration *bulkfill flowable* together is a good combination to reduce leakage fillings edge composites.<sup>12</sup> According to Tsujimoto *et al*, the *pre etching* stage before the application of *universal bonding* results in smaller edge leakage compared to *universal bonding* applications without *pre-etching*.<sup>13</sup>

Based on some of the above research descriptions, it can be concluded that the use of *adhesive bonding* affects the edge leakage of composite resin spills. The diversity of the results of the above studies is also the reason why this study was conducted to determine the type of *adhesive bonding* that is better able to reduce the leakage of composite spills.

## MATERIALS AND METHODS

This type of research is *in vitro* laboratory experimental research the *post-test only control group design*. 27 bovine incisors were cleaned and then randomly divided into 3 groups. All samples were prepared on a cervical section with a cylindrical shape of 2mm diameter and 2mm depth. Group I is a control group which means composite restoration is cured without treatment, both bonding (Tetric N Bond Universal (Ivoclar Vivadent, Zurich, Germany)), etching, or immersion of *Coca Cola*. Group II is a *self-etch* group so that *universal bonding* is directly applied with a *microbrush* without etching stage, direct bonding is applied 1 time by using a *microbrush*, then dried with *three-way syringe*, then irradiated from the occlusal direction with light cured for 20 seconds. Group III is a *total-etch* group so that it begins with etching application with a *microbrush* for 15 seconds then rinsed for 10 seconds using *three way syringe* then dried with a *cotton pellet*, bonding is applied once with a *microbrush*, then dried with *three way syringe*, then

irradiated with *lightcured* for 20 seconds from the occlusal direction. Groups II and III were then stuffed with flowable bulkfill composite resin directly applied as deep as 2 mm and light cured from the occlusal direction for 20 seconds. After all the samples were restored, *thermocycling* was carried out using a beaker and waterbath filled with water at a temperature of 5 °C and 55 °C, the treatment was carried out for 1 minute with 200 turns.

All apical parts of the sample were coated with *dental wax*. Group II and III followed by immersion in *Coca Cola* for 7 days at room temperature, immersion performed for 5 minutes with the repetition of 10 times a day (one day is considered 12 hours) when not immersed samples were stored in *Distilled water*. All samples were painted with *nail varnish* on all teeth except 1mm around the restoration, then immersed in 0.5% *methylene blue* for 24 hours, then all samples were cut buccolingually. Evaluation of edge leakage is seen from the penetration of fluid between the marginal of the restoration with the tooth substrate, seen through a stereomicroscope with magnification 8 times and observed by 3 people. Test data analysis using *Kruskall-Wallis* and *Mann-Whitney*.

## RESULTS

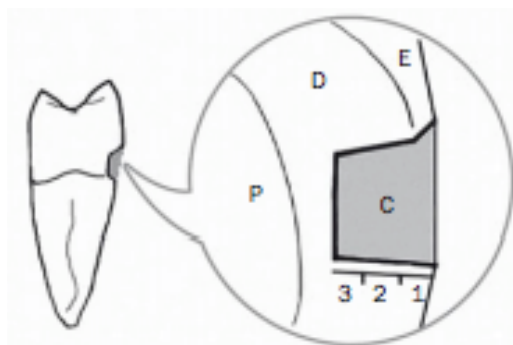
The results of measurements of leakage edge 3 groups of samples of filled with composite resin *bulkfill flowable* with *universal bonding* technique of *self-etch* and *total-etch*, after soaking with *coca cola* for 5 minutes with the repetition of 10 times per day until the seventh day, it can be seen from the penetration of liquid *methylene blue* 0.5% at the edges of the composite resin using stereomicroscopes.

Penetration of 0.5% *methylene blue* is measured by scoring. Scoring to measure the penetration depth of 0.5% methylene blue liquid at the edge of the composite restoration is the scoring determination according to Yuan *et al.*, 2015.

Based on the results of research on leakage edge flowable bulkfill composite composite shows differences in the control group and the treatment group (*self-etch* and *total-etch*). The data shows the average number of leakage scores in the control group = 3.0000, in the *self-etch* group a mean of 2.1111 was obtained, and in the *total-etch* group a mean value of 0.8889 was obtained.

The results of the study in the *self-etch* and *total-etch* groups showed a lower mean number compared to the control group and in the *total-etch* group showed the lowest mean compared to the control and *self-etch* groups. This proves that the bonding system can reduce the rate of microleakage on *bulkfill flowable* resin composite marginal restoration and the *total-etch* bonding system has the lowest mean score which shows the lowest leakage rate of composite spill edges.

This study uses the *Kruskall-Wallis* test to see significant differences. Based on the results of tests that have been done the data show significant results, namely  $P = 0.002$  ( $P < 0.05$ ). Then continued with *Mann-Whitney* test to determine differences between groups.



**Figure 1.** Microleakage evaluation of occlusal margin and gingival margin<sup>14</sup>: P (pulp), D (dentin), E (enamel), C (Cavity) edge. 0 = no penetration of color; 1 = color penetration to less than the edge of the wall; 2 = color penetration to more than the edge of the wall of the restoration; 3 = color penetration to more than the edge of the wall of the restorariion.

**Table 1.** Average results and standard deviations of flowable bulkfill composite edge leakage scores

Group	$\bar{x}$	N	Std. Deviation
control	3.000	9	.000,000
self-etch	2.1111	9	1.16667
total-etch	0.8889	9	1.05409
Total	2.0000	27	1.24035

In Table 2 the Mann-Whitney test results show that there is a significant difference ( $P < 0.05$ ) between the control group and the *self-etch* group, the control group with the *total-etch* group, and the *self-etch* group and the *total-etch* group.

## DISCUSSION

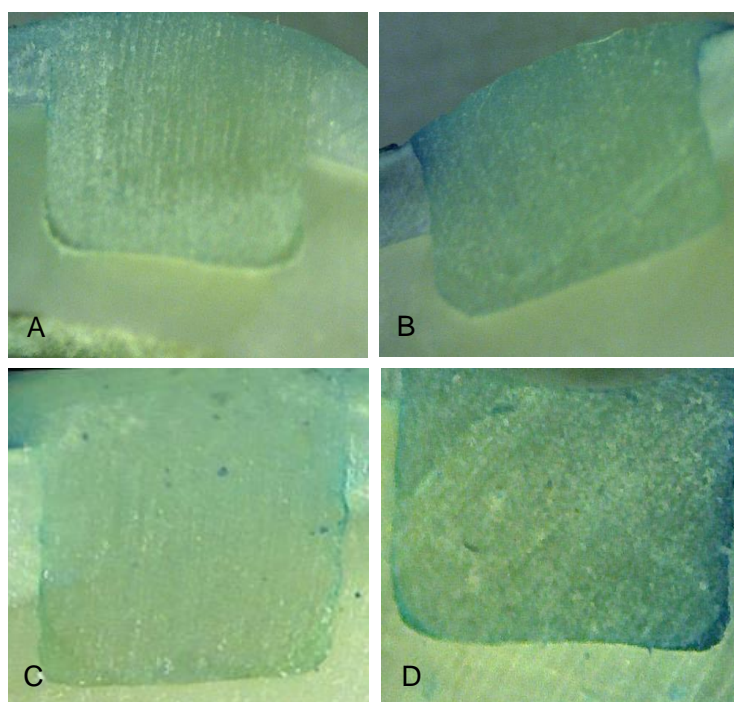
*Bulkfill* composite resin is a new composite resin that is designed as an *incremental* engineering solution and is designed to accelerate the application process of composite resins. <sup>2</sup>*Bulkfill* composite resins do not require the application of *multi-layers* because *bulkfill* allows composite resin to be clumped 4 mm at a time and minimize polymerization shrinkage. <sup>2, 6, 7</sup>

The effect of shrinkage of polymerization due to irradiation of composite resins can be reduced by the

**Table 2.** Mann-Whitney statistical test results

	Control	Self	Total
Control		0.029*	0.000*
Self			0.038*
Total			

\* = Significant



**Figure 2.** (A) no penetration of methylene blue with score 0; (B) methylene blue penetration less than 1/3 depth of restoration with score 1; (C) methylene blue penetration reach 2/3 depth of restoration with score 2; (D) methylene blue penetration more than 2/3 depth of restoration.

addition of adhesive bonding because the bonding system aims to provide a layer that is capable of binding strongly enough between the composite restoration and tooth structure to resist mechanical forces and stresses due to the process of polymerization. The bonding system is composed of etching, primers, adhesive particles, solvents, initiators and filler particles.<sup>1,9</sup>

The bonding system has evolved both regarding the composition and mechanism of action in dental tissue, as well as its components and the number of clinical steps required for application. This allows for the achievement of lower technical sensitivity and an equivalent level of performance in enamel and dentin. The latest bonding system is currently called *universal bonding*. The application of *universal bonding* can be done by the method of *self-etch* or *total-etch*.<sup>1,13,15</sup>

The *total-etch* bonding system begins with a separate etching procedure using 30-40% as an phosphate which can eliminate the<sup>1,8,15</sup> *smear layer* so that the inter tubular surface of the demineralized dentin which results in an open collagen husk and the formation of micro porosity is due to the etching procedure of the *total-etch process* long so that it causes a high potential for postoperative hypersensitivity.<sup>8</sup>

In the *self-etch* system, there is no etching procedure first. This causes a pH that is not too acidic so *self-etch* does not cause dentin demineralization.<sup>1,8</sup> *Self-etch* also has a disadvantage, which has not strong attachment to enamel. *Self-etch* only binds superficially to the enamel and dentin and almost does not remove the remaining hydroxyapatite crystals (*smear layer*) but binds to the *smear layer* to form a *hybrid bond* 1, 8, 10, 16.

The above theory is the basis of this study to determine whether there are differences in the value of *bulkable flowfill* composite edge leakage from each bonding system technique both *self-etch* and *total-etch*.

The sample teeth used in this study were bovine teeth that had been treated soaked in cola for 5 minutes 10 times daily treatment (one day assumed for 12 hours) to 7 days, this time was based on research conducted by Narsimha (2011).<sup>17</sup> The calculation of the day assumes that each person who drinks Coca-Cola is assumed to take about 5 minutes and this is done to ensure a realistic consumption pattern and resembles the exposure of coca cola to daily life. Then after being converted, this research is assumed to be equivalent to 100 days of consumption rounded up to 3 months.<sup>18</sup>

This timing refers to a study conducted by Aprilia *et al.*, 2007, which assumed immersion for 1 week (7 days) is equivalent to (7 days x 12 hours x 60 minutes) divided by 5 minutes x 10 times treatment, equal to 100 days, this means the same as 3 months of use.<sup>18</sup>

Coca-Cola has a low pH, ie 2.53 (below 5.5) that is said to be critical pH in the oral cavity as hydroxyapatite soluble below pH 5.5<sup>4,5,17</sup> and because the teeth are composed of hydroxyapatite, the tooth will be decalcification dental materials which will be dissolved by saliva that would

cause demineralizes. This is illustrated by the following formula:<sup>5</sup>



The more acidic a product of food / beverage,  $\text{H}^+$  will continue to rise so  $6\text{PO}_4^{3-}$  will turn into  $\text{H}_2\text{PO}_4^{2-}$  and ion  $\text{OH}^-$  will dissolve in water so that over time will participate dissolved calcium.<sup>5</sup>

In this study, the edge leakage value of *flowable bulkfill* composite resin is characterized by the penetration of *methylene blue dyes* which will be assessed by scoring assessment according to Yuan *et al* (2015).<sup>14</sup> Penetration of dyes is observed with a digital stereomicroscope.

The scoring system is used because exact measurement of the depth of direct paint penetration is not possible, so the results are grouped in the score category. Scores are ordinal data and then we use rank-based nonparametric tests whose purpose is to determine whether there are statistically significant differences between the three groups of variables on a numerical and ordinal scale. This study uses 3 groups so the *Kruskall-Wallis* test must be conducted first to determine whether there is significance between groups. The results obtained from the *Kruskall-Wallis* test result indicate that there are significant differences between the samples so the *Mann-Whitney* test can be continued to determine the median value and differences in the median values of the three groups so that it can be seen which group has the least leakage of the edges.

After the median value of each group is known, then do one group comparison to another group one by one, starting comparing the control group (group 1) with the *self-etch* group (group 2), there is significance between groups, then comparing group 2 and *total group etch* (group 3), there was also significance. Significance can be deduced from the value of  $p = 0.005$ . The significance value makes  $\text{H}_0$  rejected and  $\text{H}_1$  accepted. This is consistent with the initial hypothesis of the study.

In this study, the results of the degree of edge leakage in the sample group using the *total-etch* technique are smaller than the sample group using the *self-etch* technique and the sample group that does not use the bonding system or the immersion of *coca cola* and *thermocycling*. This is caused by: first, clean substrate surface of the *smear layer* in the *total-etch* technique and also increase it which is without the exposure of hydroxyl enamel group to hydroxyapatite which causes the substrate to become hydrophilic so that the *wettability* and *surface energy* increase so that it will result in penetration of the adhesive monomer of the bonding material to be easier and stronger because micromechanical *interlocking* occurs and increases marginal adaptation so that the edge leakage that occurs is smaller than the *self-etch* or control groups. Second, *resin tags* group of *self-etch* formed between the *smear layer* with MDP monomer form of *smear plugs* so that the resin tags were weaker than resin tags in the group *total-etch* formed between monomer MDP directly with hydroxyapatite on the tooth. Third, although the control group was not treated with *coca cola* soaking

the edge leakage persisted and was even greater than the *self-etch* group due to the absence of a bonding system resulting in a significant *gap* between the restorations with the tooth substrate due to composite shrinkage which is the effect of polymerization during irradiation.

The presence of a *smear layer* has a profound effect on the degree of restoration edge leakage. Based on the experimental results, the *total-etch* technique provides a lower edge leakage compared to the *self-etch* system. This lower edge leakage allows the higher final success of composite restorations than *self-etch* systems.

Universal bonding was developed with a view to meeting the demands of dentists in accordance with the preferences of each clinician's restoration technique both self-etch and total-etch. This bonding material is contained by a phosphate group monomer, the MDP-10 monomer.<sup>15,19</sup> MDP-10 has a dihydrogenphosphate group and a carbonyl chain. The dihydrogenphosphate group has a function for chemical etching and binding, while the long carbonyl chain gives hydrophobic properties and hydrolytic stability to these acid monomers. MDP will also interact with hydroxyapatite in teeth to form bonds.<sup>15,19</sup>

However, good and stable chemical bonds are not enough; stable chemical bonds must also be followed by good micromechanical bonds between the substrate and teeth. This requires the formation of microporosity that is consistent in shape and has sufficient depth so that it has good retention.<sup>15,19</sup> The etching process occurs because the MDP-10 monomer is acidic (pH = 2.7) but not as strong as 37% phosphoric acid (pH = 0.5). Phosphoric acid 37%, is the etching substance most often used to produce consistent microporosity without injuring the pulp, while the etching process with weak acids will result in microporosity that is formed irregularly and has insufficient depth, this causes inadequate micromechanical retention and causes *debonding* and edge leakage are prone to occur as a result of debonding.<sup>1</sup> The results of this experiment confirm the theory, the use of etching before bonding application will increase the retention strength of the restoration and substrate, because in addition to MDP-10 particles that form stable chemical bonds with HAp, it is also aided by micromechanical bonds with microporosity formed in dentin.

## CONCLUSION

Leakage edge *flowfill bulkfill* composite restoration with *total-etch* technique and *self-etch* technique is smaller than the control group, and edge leakage *flowable bulkfill* composite restoration with *total-etch* technique is smaller than the *self-etch* group .

## REFERENCES

1. Anusavice K, Shen C, Rawls H. Phillips' science of dental materials. 12th ed. Elsevier; 2013.

2. Permana D, Sujatmiko B, Yulianti R. Comparison of micro-resin composite bulk-leak leakage rates with incremental and bulk oblique filling techniques. Indonesian Dentistry Magazine. 2016 ; 2 (3): 135.
3. Dharmawati I. Consumption Soft Drink resulting to damage to dental. Journal of Nutrition . 2015 [cited 29 November 2019] ; 6 (1): 43-50. Available from: <http://www.poltekkes-denpasar.ac.id/wp-content/uploads/2017/12/dharmawati-JIG-v6n1-February-2015.pdf>
4. Prasetyo E. The acidity of soft drinks decreases tooth surface hardness [Internet]. Journal.unair.ac.id. 2005 [cited November 29, 2019]. Available from: <http://journal.unair.ac.id/filerPDF/DENTJ-38-2-04.pdf>
5. Borjian A, Ferrari C, Anouf A, Touyz L. Pop-Cola Acids and Tooth Erosion: An In Vitro , In Vivo, Electron-Microscopic, and Clinical Report. International Journal of Dentistry. 2010 ; 2010: 1 -12.
6. Monterubbianesi R, Orsini G, Tosi G, Conti C, Librando V, Procaccini M et al. Spectroscopic and Mechanical Properties of a New Generation of Bulk Fill Composites. Frontiers in Physiology. 2016 ; 7 .
7. Laurensia E, Untara T, Daradjati S. Differences In Micro Leakage Results of Flowable Bulkfill Composite Composites Using Total-Etch and Self-Etch Bonding Materials. J Ked Gi [Internet]. 2014 [cited 29 November 2019] ; 5 (2): 57-61. Available from: <https://journal.ugm.ac.id/jkg/article/view/27837>
8. Yousaf A, Aman N, Manzoor M, Shah J, Dilrasheed. Postoperative sensitivity of self-etch versus total-etch adhesive. J Coll Physicians Surg Pak [Internet]. 2014 [cited 29 November 2019] ; 24 (6). Available from: <https://www.ncbi.nlm.nih.gov/pubmed/24953909>
9. Meerbeek B, Munck J, Yoshida Y, Inoue S, Vargas M, Vijay P et al. Adhesion to Enamel and Dentin: Current Status and Future Challenges [Internet]. Pdfs.semanticscholar.org. 2003 [cited November 29, 2019]. Available from: <https://pdfs.semanticscholar.org/a85d/cf9565a49580260ba7a9b1a8cb810f452f62.pdf>
10. Sundari I, Triaminingsih S, Soufyan A. Adhesion Strength of Resin Composite Restoration on Dentin Surface with Self-Etch Adhesive System in Various Temperatures. Journal of Indonesian Dentistry. 2008 ; 15 (3).
11. Burgess J, Sadid-Zadeh R, Cakir D, Ramp L. Clinical Evaluation of Self-etch and Total-etch Adhesive Systems in Noncarious Cervical Lesions: A Two-year Report. Operative Dentistry. 2013 ; 38 (5): 477-487.
12. Bolgöl B, Çelenk S, Kiliç G, Seker O, Simsek I, Ayna B. Leakage testing for different adhesive systems and composites to permanent teeth. Nigerian Journal of Clinical Practice. 2015; 0 (0): 0.
13. Tsujimoto A, Barkmeier W, Takamizawa T, Latta M, Miyazaki M. The Effects of Phosphoric Acid Pre-etching Times on Bonding Performance and Surface Free Energy with Single-step Self-etch Adhesives. Operative Dentistry. 2016 ; 41 (4): 441-449.
14. Yuan H, Li M, Guo B, Gao Y, Liu H, Li J. Evaluation of Microtensile Bond Strength and Microleakage of a Self-Adhering Flowable Composite. Journal of Adhesive Dentistry [Internet]. 2015 [cited 29 November 2019] ; 17 (6): 538. Available from: <https://www.ncbi.nlm.nih.gov/pubmed/26734678>
15. Burke M, Lawson A, Green D, Mackenzie L. What's new in dentin bonding ?: Universal adhesives. Dental Update [Internet]. 2017 ; 44 (4): 328-340. Available from: <https://>

- fgdpScotland.org.uk/wp-content/uploads/2016/05/Dent\_Update\_2017\_Dentine-Bonding.pdf
16. Muñoz M, Luque I, Hass V, Reis A, Loguercio A, Bombarda N. Immediate bonding properties of universal adhesives to dentine. *Journal of Dentistry*. 2013 ; 41 (5): 404-411.
  17. Narsimha V. 2011. *People's Journal of Scientific Research* [Internet]. 2019 [cited 29 November 2019] ; 4 (2): 34-39. Available from: [https://www.pjsr.org/July11\\_pdf/8-Vanga%20Narsimha.pdf](https://www.pjsr.org/July11_pdf/8-Vanga%20Narsimha.pdf)
  18. Aprilia A, Rochyani L, Rahardianto E. Effect of Coffee Drinks on Color Changes in Composite Resin [Internet]. 2007 [cited November 29, 2019]. Available from: Effect of Coffee Drinks on Color Changes in Composite Resins | Aprilia | *Journal of Indonesian Dentistry*
  19. Giannini M, Makishi P, Ayres A, Vermelho P, Fronza B, Nikaido T et al. Self-Etch Adhesive Systems: A Literature Review. *Brazilian Dental Journal*. 2015 ; 26 (1): 3-10.