AN IN -VITRO STUDY TO EVALUATE THE DENTIN BOND STRENGTH AND COLOR CHANGE OF BULK FILL RESIN COMPOSITES WITH DIFFERENT INCREMENTAL THICKNESS USING TWO CURING MODES

Raafat. F. Alshora* Nady I. Mohammed** *and* Ahmed A. Abd El-Ghany***

ABSTRACT

This study was directed to evaluate the dentin bond strength and color change of bulk fill resin composites with different incremental thickness using two curing modes. Extracted human molars teeth were used to prepare one hundred and twenty specimens. For shear bond strength, the specimens were divided into two main groups according to types of composite (Tetric N Ceram, Quixfil). Each main group was divided into two groups according to thickness of composite (2& 4 mm). Each group was divided into two sub-groups according to curing mode (Continuous and soft start mode). Then, each group was divided into three classes according to storage time (one day, three months, and six months). Fr color change, one hundred and twenty discs of resin composite were used in this study. The discs were divided into two main groups according to types of composites (Tetric N Ceram, Quixfil). Each main group was divided into two groups according to thickness of composite (2 mm, 4 mm). Each group was divided into two sub groups according to curing modes (Continuous and soft start mode). Then, each sub-group was divided into three classes according to immersions media (Acidic ph, alkaline ph solution. Neutral ph). Finally, each class was divided into three sub classes according to storage time (one day, three months, and six months). The shear bond strength was evaluated using universal testing machine at a crosshead speed of 0.5 mm/min until failure occurred. Data were recorded, tabulated and statistically analysed. The specimen's colors were measured using a Reflective spectrophotometer. The result showed the highest bond strength was recorded by (Quixfill, 4 mm, soft start at one day). For color change the (Quixfil, 4 mm, soft start, and acidic solution recorded the highest color change.

INTRODUCTION

In the last few years, aesthetic concerns have produced an increased demand for tooth-colored restorations, even in posterior teeth [1]. A new category of bulk fill resin composite has been introduced over the past few years. They were developed to speed up the restoration process by enabling up to 4 mm thick increments to be cured in a single step[2]. Different methods had been introduced for composite resin insertion techniques including incremental and bulk fill techniques[3]. Even though incremental layering may be necessary to ensure adequate polymerization of the composite resin, there are also some disadvantages to this technique, air entrapment between the different layers May occur[4]. The controversy among researchers and practitioners with regard to the appropriate placement technique, namely, bulk placement that offers much greater depth of cure [5].

Bulk fill resin composite is a light-cured composite material that can be placed and cured in bulk increments of 4 – 5 mm thickness[6]. Incremental layering has long been accepted as a standard technique for placement of resin composite in cavity preparations. Typically, this technique consists of placing increments of resin composite material in a thickness of 2 mm or less followed by exposure to light curing from an occlusal direction and

*Demonstrator of Operative Dentistry Department, Faculty of Oral Medicine, Assuit, Al-Azhar University.
**Assistant Professor of Operative Dentistry Department, Faculty of Oral Medicine, Boys, Cairo, Al-Azhar University.
***Lecturer of Operative Dentistry Department, Faculty of Oral Medicine, Assuit, Al-Azhar University..

then repeating increments until the preparation is filled [7]. One obvious advantage for the incremental technique is the limitation of the thickness of resin, which provides adequate light penetration and subsequent polymerization that results in enhanced physical properties, improved marginal adaptation and decreased cytotoxicity of the resin composite [8]. Another reason to use the incremental technique is to decrease the amount of shrinkage occurring during polymerization, which is beneficial because the developing stress can cause cuspal deformation with resulting sensitivity or micro cracks in resin or tooth structure. The stress can also cause adhesive failure at the tooth/resin interface resulting in marginal gap, micro leakage, and secondary caries [9]. Despite these benefits, the incremental technique has disadvantages, that may include; the possibility of incorporating voids or contamination between composite layers, bond failures between increments, difficulty in placement because of limited access in conservative preparations, and the increased time required to place and polymerize each layer [10]·To overcome

the problem from incremental techniques there are new categories of resin composites that have been produced which can be successfully packed in bulk and cured in thicknesses of five mm while the maximum recommended thickness for most commonly used composites is two mm[11]. The color stability of composite is an important factor for esthetic demand, however the increasing of incremental thickness may affect color stability, the bulk-fill resin composites exhibit different optical properties including excellent translucent compared with regular resin composites[12].

MATERIAL AND METHODS

Measurement of shear bond strength:

One hundred and twenty sound human molar teeth free from caries the teeth were randomly divided into two main groups according to types of composite Tetric N Ceram and Quixfil bulk. Each main group was divided into two groups according to thickness of composite 2 mm, and 4 mm thick-

Table (1) Materials used in the study:

Materials	Specification	Composition	Manufacturer	Batch Numb er
Tetric NCe- ram Bulk fill	Nano hybrid bulk fill composite	Dimethacrylates 21%, Polymer filler 17%, (Barium glass filler, Ytterbium trifluoride, Mixed oxide 61%), (Additive, Initiators, Stabilisers, Pigments < 1%). (Bis-GMA*, UDMA**)	Ivoclar Vivadent AG FL-9494 Schaan/ Liechtenstein, USA. http:www.ivoclarviva- dent. com.	27917
Quixfill Bulk fill	Microhybrid bulk fill composite	-Bis-EMA *** ,UDMA -TEGDMA+,TMPTMA++- Butane-1,2,3,4tetracarboxylic acid -photo initiator camphor quinone - Accelerator dimethyl amino benzoic acid ethyl ester	DENTSPLY Detrey GmbH Germany	15090 951
Tetric N Bond	Bonding system	Phosphoric acid acryl ate Bis –GMA HEMA+++	Ivoclar Vivadent AGFL- 9494 Schaan/ Liechtenstein, USA	34551
Eco- Etch (etchant)		37% Phosphoric acid gel	Ivoclar Vivadent AG FL-9494 Schaan/ Liechtenstein, USA	

ness. Each group were divided into two sub groups according to curing modeContinuous mode and soft start mode. Then, each group was divided into three classes according to storage timeone day,three months, and six months. The occlusal third of each tooth was cut off using a diamond disk under cooling, therefore exposing a flat dentin surface. In order to get the standardized deep dentin level a flat end cylindrical bur with predetermined 2mm mark used to removal half of bucco-lingual dimension of the flat occlusal surface measured by digital caliper. A standard smear layer was created using water cooled sand papers. The dentin surface was rinsed with water. Following the adhesive procedures, a composite resin blocks were built using bulk fill technique (4 mm) on the occlusal direction using specially design mold. After curing each tooth was mounted on the cutting machine. The ground dentin surfaces of the samples were air-dried and for each specimen, the bonding area of the dentin surface was then treated with the three-step etch-and-rinse adhesive system. The samples was etched for 15 sec with 37% of phosphoric acid etch* and rinsed for 20 sec with water, and bonded according to manufacturer's instructions, all samples were polymerized for 20 sec using LED curing unit with two curing modes that have a light output of 1000 mW\cm2 according to the manufacturer.After curing of adhesive system, a split Teflon mold as described above (inner diameter: 2.5 mm and have two heights: 2mm, and 4mm) was clamped to the dentin surface of each sample and the respective mold was filled in bulk with one of the two bulk fill composite. The composite was made flush with the mold by use of a glass slide and excess resin was removed. Then the composite was cured with two curing modes (continuous and soft start modes) for each group. All samples were stored in distilled water at 37 C in incubator to regulate the aging temperature 37 C either for one day, three months or six months. The storage media was changed every week for all the aged specimens

Shear bond strength measurements:

After the water storage for each group, all samples were individually and vertically mounted on a computer controlled materials testing machine* (Model 3345; Instron Industrial Products, Norwood, USA) with a load cell of 5 kN and data were recorded using computer software (Bluehill Lite; Instron Instruments). The samples were secured to the lower fixed compartment of testing machine by tightening screws. Shearing test was done by compressive mode of load applied at tooth resin interface using a mono beveled chisel shaped metallic rod attached to the upper movable compartment of testing machine travelling at crosshead speed of 0.5 mm/min. The load required to debonding was recorded in Newton.

Measurement of color change

One hundred and twenty disc of resin composite were used in this study, the discs were randomly divided into two main groups according to types of resin composites Tetric N Ceram and Quixfil. Each main group was divided into two groups according to thickness 2mm, and 4mm thickness. Each group was divided into two sub groups according to curing modes Continuous mode and soft start mode. Then each sub group was divided into three classes according to immersions media Acidic ph solution, alkaline pH, neutral ph. Finally, each class was divided into three sub classes according to storage time one day, three months, and six months. All materials in this study were used according to the manufacturer's instructions, for color measurement two type of bulk fill resin composite (Tetric N Ceram and Quixfil), two thicknesses of bulk fill composite (2mm and 4mm), and two curing mode (continuous mode and soft start modes) LED light curing unit were used. The teflon mold hold on glass slap and the celluloid strip was placed under the mold to prevent the composite from sticking in glass slap. Then the each type of bulk fill composite was packed in

each mold (2mm, and 4mm) then the composite was covered by another glass slap to flush the surface of composite and flattened the surface, all specimens were polymerized according to the manufacturer's instructions using LED light curing unit. The discs were removed from teflon mold and measured using a reflective spectrophotometer. During storage time the all specimen stored in distilled water thereafter preparation of immersions media and measuring the PH of each solution by ph meter, the all specimen were divided into three equal groups, where group1 for acidic pH media (Pepsi), group2 for alkaline pH media (Ginger), and group3 for neutral pH media (distilled water). During the storage time the specimens immersed in acidic and alkaline ph solution for period of 20 minute per day and returned again to distilled water, this cycle was repeated regularly every day up to the end of storage time (one day, three months and six months) at 37C in incubator to regulate the aging temperature 37C.

Color measurement

The specimens colors were measured using a reflective spectrophotometer*. The aperture size was set to 4 mm and the specimens were exactly aligned with the device. A white background was selected and measurements were made according to the CIE L*a*b* color space relative to the CIE standard illuminant D65. The color changes (ΔE) of the specimens were evaluated using the following formula:

$$\Delta E_{CIELAB} = (\Delta L*2 + \Delta a*2 + \Delta b*2)^{\frac{1}{2}}$$, Where: L* = lightness (0-100), a* = (change the color of the axis red/green) and b* = (color variation axis yellow/blue).

RESULTS

I. Evaluation of shear bond strength

Shear bond strength of Quixfill composite group recorded statistically significant ($p<0.05$) higher shear bond strength mean values (13.244) when compared with Tetric N Ceram composite group, Shear bond strength of 4mm group curing depth group recorded statistically significant ($p<0.05$) higher shear bond strength mean values (11.388) than 2mm group curing depth mean value (9.395).,Evaluation of shear bond strength revealed that the soft start curing mode group recorded statistically non-significant ($p>0.05$) higher shear bond strength mean values (10.49) than continuous curing mode group, Evaluation of shear bond strength revealed that the one day storage time group record-

| Variables	2mm						4mm					
	Continuous			Soft			Continuous			Soft		
	One day	3m	6 m	One day	3m	6 m	One day	3m	6 m	One day	3m	6 m
Tetric N Ceram	9.01 ±1.9	7.09 ±3.1	3.94 ±.56	6.80 ±0.73	3.11 ±0.68	5.74 ±0.65	13.28 ±0.1	9.34 ±1.6	7.21 ±2.1	10.61 ±0.51	9.06 ±1.1	5.80 ±0.2
Quix-Fill	17.75 ±3.4	11.29 ±1.4	7.65 ±1.8	17.63 ±2.2	13.98 ±2.4	8.77 ±0.75	18.50 ±1.3	11.54 ±1.5	9.30 ±0.7	17.03 ±3.2	14.04 ±0.5	11.46 ±1.1
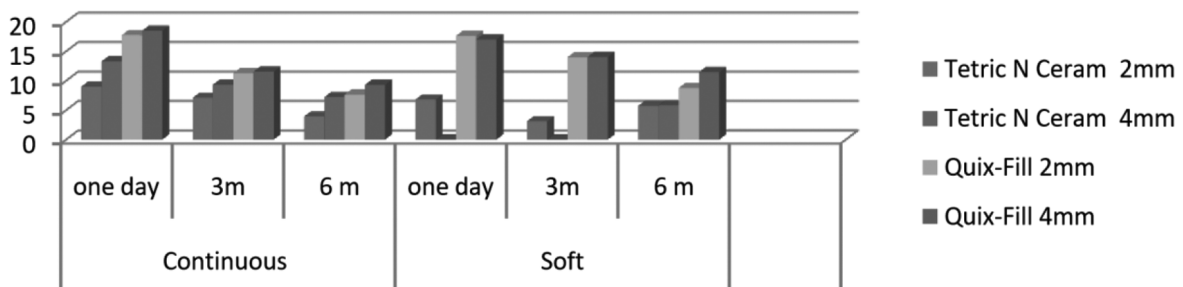
ed statistically significant (p<0.05) highest shear bond strength mean values (13.625), followed by 3 months storage time group mean values (10.067), while the lowest statistically significant (p<0.05) shear bond strength mean values recorded after 6 months storage time group (7.48).

II. Result for Color change

Evaluation of color change of bulk fill composite revealed that the Quixfill composite group recorded statistically significant (p<0.05) higher color change mean values (11.114) when compared with Tetric N Ceram composite. The 4mm group curing depth recorded statistically significant (p<0.05) higher color change mean values (12.14) than 2mm group curing depth When evaluation of color change of two composites, it was found that Soft start curing mode group recorded statistically non-significant (p>0.05) higher color change mean values (10.238) than Continuous curing mode group. It was found that acidic solution subgroup recorded statistically significant (p<0.05) highest color change mean values (12.402), followed by alkaline solution sub-group mean values (11.868), while the lowest statistically significant (p<0.05) color change mean values recorded with neutral solution subgroup (4.463). Measurement of color change showed that the 6 months storage time group recorded statistically significant (p<0.05) highest color change mean values (11.323), followed by 3 months storage time group mean values (10.797), while the lowest statistically significant (p<0.05) color change mean values recorded after one day storage time.

Variables		2mm						4mm					
		Continuous			Soft			Continuous			Soft		
		One day	3m	6 m	One day	3m	6 m	One day	3m	6 m	One day	3m	6 m
Acidic	Tetric N Ceram	1.5 ±0.1	1.7 ±0.7	7.6 ±3.7	5.9 ±3.1	5.4 ±3	6.1 ±1.4	1.4 ±0.5	13.5 ±2.7	17.0 ±8	8.5 ±2.6	11.3 ±3.4	15.6 ±6.8
	Quix-Fill	14.0 ±3.2	15.1 ±3.5	14.7 ±5.3	12.1 ±0.6	17.5 ±1.1	5.7 ±2.2	13.8 ±1.3	24.5 ±1.4	25.2 ±5.4	24.1 ±8.8	17.8 ±1	17.8 ±1.3
Alkaline	Tetric N Ceram	2.4 ±0.2	8.8 ±2	7.9 ±2.6	4.8 ±0.5	12.0 ±5.7	14.8 ±1.1	10.4 ±1.6	14.9 ±1.7	16.3 ±0.6	5.5 ±2.2	16.3 ±5.1	20.7 ±2.1
	Quix-Fill	4.7 ±0.9	13.4 ±3.7	7.9 ±2.5	4.5 ±1.3	13.2 ±1.2	13.9 ±1.7	7.5 ±2	19.1 ±2.2	10.7 ±0.2	11.9 ±2.8	22.2 ±2.2	20.9 ±3.9
Neutral	Tetric N Ceram	1.5 ±0.2	7.8 ±2.7	4.7 ±0.3	4.1 ±0.8	4.3 ±1.8	3.6 ±0.15	6.6 ±1.8	3.9 ±1.2	1.9 ±0.4	3.3 ±1	8.7 ±0.2	8.7 ±2.7
	Quix-Fill	1.3 ±0.3	1.6 ±0.2	3.9 ±1.8	1.6 ±0.1	1.3 ±0.06	1.5 ±0.25	1.9 ±0.3	6.6 ±1.9	5.3 ±1.6	5.5 ±2	10.8 ±2.9	6.8 ±2.3
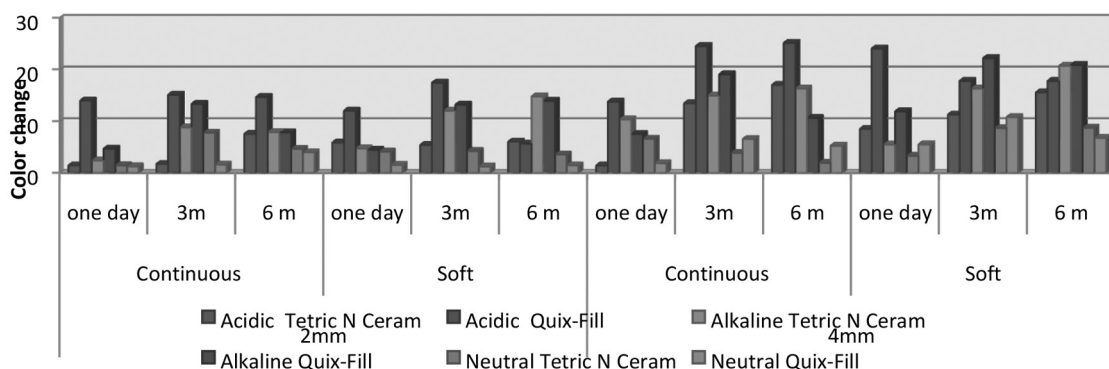
DISCUSSION

The data in results showed that the Quixfill composite have significant higher bond strength than the Tetric N Ceram with all storage time. This is probably due to the effect of the different filler systems, the filler volumes of these materials. The bigger filler size of Quixfill decreases the total filler surface, consequently, the filler matrix interface, and reducing refractive index play a crucial role in achieving higher depth of cure which improves the degree of conversion, which improve the bond strength[13].

This confirmed by Oznurhan et al[14], whose found that the size of the filler particles of these materials may have an effect on their bond strength. This finding disagree with Alrahlah et al[17] who reported that Tetric N ceram had the greatest depth of cure amongst the bulk fill composites because of the particles are smaller than the wavelength of light and cause minimal or zero scattering of photon. The data showed that the 4mm group cure depth recorded statistically significant higher shear bond strength mean values than 2mm group cure depth.

This is probably due to the modification in monomers and photo initiator systems by adding a new initiator in addition to the camphor quinone amine initiator systems that is ivocerin, that allow light to penetrate into deepest layer that improve hardness, decrease polymerization stress thus improve the bond strength. Ivocerin is described as a germanium based initiator system with a higher photo curing activity than camphor quinone amine because of its higher absorption in the wavelength region between 400 and 450 nm. This finding agrees with Flury et al (2014)[18], who exhibited the bulk fill composite have a higher depth of cure, the improvements in their initiator system, and increased translucency improve the bond strength[19]. This confirmed with different in vitro studies Ilie et al (2013)[20] that re-

vealed the bulk fill have high mechanical properties that cured in larger increments, as the degree of cure and bond strength can be maintained within 4mm layers at an irradiation time of up to 20 s. This finding disagree with Benetti et al[21] who exhibited a depth of cure of bulk fill composite was statistically similar to that of the conventional resin composite.

The data showed that the soft curing mode recorded statistically higher shear bond strength mean values than Continuous curing mode group. This may be due to soft-start polymerization mode reduce the initial conversion, and control the flow of composite resin.

This finding was confirmed by Barros et al[22] who showed that the reduction of this initial speed can be obtained through the use of low intensity units that produce few free radicals. This lower speed gives the composite more time for molecular rearrangement, reducing the stress caused by polymerization shrinkage[23].

This finding is in disagreement with Price et al[24], who suggested the stepped photo-polymerization system offers no advantages over a single high intensity curing. It is particularly interesting given that there is a difference of approximately 20% in energy output between the single cure and stepped systems. It appears that small difference in energy output is not of sufficient magnitude to cause large differences in shear bond strength. Data showed that the one day storage time recorded statistically significant highest shear bond strength followed by three months storage time while the lowest statistically significant shear bond strength recorded after six months storage time. This decrease in bonding effectiveness by time might be explained by degradation of interface components by water storage. This finding confirmed that the bond strength is descending from 24 h to 6 months of water storage.

REFERENCES

1. Demarco FF, Correa MB, Cenci MS, Moraes RR, Opdam NJM. Longevity of posterior composite restorations: not only a matter of materials. Dent Mater. 2012; 28:87-101.

2. Sakaguchi RL, Douglas WH, Peters MC. Curing light performance and   polymerization of composite materials J Dent. 1992 ; 20:183–88.

3. Ilie N, Hickel R. Investigations on a methacrylate-based flowable composite based on the SD technology. Dent Mater 2011; 27: 348-55.

4. Sakaguchi RL, Douglas WH, Peters MC. Curing light performance and   polymerization of composite restorative materials. J.Dent 1992; 20:183 - 88.

5. Abbas G, Fleming GJP, Harrington E, Shortall ACC, Burke FJT. Cuspal movement and micro leakage in premolar teeth restored with a packable composite cured in bulk or in increments. J Dent 2003; 31: 437- 44.

6. Lazarchik DA, Hammond BD, Sikes CL, Looney SW, Rueggeberg FA. Hardness comparison of bulk filled Transtooth and incremental filled occlusal irradiated composite resins. J. Prosthet Dent. 2007; 98:129 - 40.

7. Giachetti L, Scaminaci Russo D, Bambi C, Grandini R. A review of polymerization shrinkage stress: current techniques for posterior direct resin restorations. J Contemp Dent Pract. 2006;7: 79 - 88.

8. Podshadley A, Gullett C, Crim G. Interface seal of incremental placement of visible light-cured composite resins. J Prosthet Dent. 1985;53: 625 - 26.

9. Goracci C, Cadenaro M, Fontanive L, Giangrosso G, Juloski J, Vichi A, Ferrari M. Polymerization efficiency and flexural strength of low stress restorative composites. Dent Mater. 2014; 30: 688 - 94.

10. Rueggeberg FA, Caughman WF, Curtis JW Jr, Davis HC. Factors affecting cure at depths within light-activated resin composites. Am, J. Dent1993; 6: 91 - 5.

11. Baharav H, Brosh T, Pilo R, Cardash H. Effect of irradiation time on tensile properties of stiffness and strength of composites. J.Prosthet Dent1997; 77: 471 -74.

12. Flury S, Hayoz S, Peutzfeldt A, Husler J, Lussi A. Depth of cure of resin composites: is the ISO 4049 method suitable for bulk fill material2012; 28:52-28.

13. Gordon's j .Christensen. Challenges of Bulk-Fill Resins January. Dent Mater. 2012; 30: 688-94.

14. Ilie N, Keßler A, Durner J. Influence of various irradiation processes on the mechanical properties and polymerization kinetics of bulk-fill resin based composites. J Dent 2013; 41:695-02.

15. Alrahlah A, Silikas N, Watts D. Post-cure depth of cure of bulk fill dental resin-composites. Dent Mater. 2014;30:149-54.

16. Ilie N, Bucuta S, Dreaenert M. Bulk-fill Resin-based Composites: an in vitro assessment of mechanical performance. Oper Dent; 2013; 38:618-25.

17. Benetti AR, Havndrup-Pedersen C, Honoré D, Pedersen MK, Pallesen U. Bulk-fill resin composites: Polymerization contraction, depth of cure, and gap formation. Oper Dent. 2015; 40:190-200.

18. Davidson CL, Feilzer AJ. Polymerization shrinkage and polymerization shrinkage stress in polymer based restoratives. J Dent. 1997; 25: 435- 40.

19. Salita YT, Dunn WJ, Peters CB. Effect of shorter polymerization times when using the latest generation of light-emitting diods. Am J Orth. Dent. 2005; 128:744- 48.

20. Asmussen E, Peutzfeldt A. Influence of pulse-delay curing on softening of polymer S structures. J Dent Res 2001; 80:1570–73.

22. Lim BS, Ferracane JL, Sakagushi RL, Condon JR. Reduction of polymerization contraction stress for dental composites by two-step light-activation. Dent Mater. 2002; 18: 436- 44.

23. Price RB, Bannerman RA, Rizkalla AS, Hall CH. Effect of stepped vs. continuous light curing exposure on bond strengths to dentin. Am J Dent 2000; 13:123-28.

24. Abdalla AI, Felzer AJ. Four-year water degradation of a total etch and two self-etching adhesives bonded to dentin. J Dent 2008; 36:611-17.