STUDY REGARDING THE INFLUENCE OF THE DISTANCE FROM LIGHT CURING UNIT TIP TO MATERIAL SURFACE AND OF THE LIGHT CURING MODE ON POLYMERIZATION EFFECTIVENESS OF A BULF FILL BASE COMPOSITE RESIN

Simona Stoleriu[1], Gianina Iovan[1*], Cristina Angela Ghiorghe[1], Irina Nica[1], Ionuț Tărăboanță[1], Antonia Moldovanu[1], Sorin Andrian[1]

„Grigore T. Popa" University of Medicine and Pharmacy Iasi, Department of Odontology-Periodontology, Fixed Prosthesis [1]

* Corresponding author: *e-mail:* gianinaiovan@yahoo.com

ABSTRACT

The aim of the study was to evaluate the influence of some characteristic of the light curing unit (polymeriyation mode and the distance between light curing tip to the surface of composite resin) on the efficiency of SDR® Plus bulk fill flowable base composite. U (universal), A2, and A3 shades was used in the study. Fiftyfive samples from each shade were prepared by injection the flowable resin in cylindrical plastic molds with the intern diameter of 5 mm. Three types of plastic molds according to their hight were used: 6 mm, 8 mm, and 10 mm. The tip of the light curing unit was placed on top of the mold and composite resin was light cure for 20 seconds for U shade and 40 seconds for A2 and A3 shades. Top and bottom surface Vickers hardness was determined using a digital microhardness. The hardness ratio of each specimen was also calculated Polymerization efficiency of SRD® Plus bulk-fill flowable base composite resin was not affected by the distance from the light sourse to the surface of the material and the light curing mode. Staggared polymerization mode of the light curing unit determines slightly low values of surface hardness when comparing to inclined and normal polymerization modes. Darker shades of bulf fill flowable base composite resin seems to present decreased surface hardness when comparing to ligther shades irrespective the distance from the light curing unit to the material surface and the mode of polymerization.

Key words: bulk-fill flowable composite, SRD® Plus, surface hardness, polymerization

Setting reaction of dental composite resides in a chemical reaction between dimethacrylate resin monomers which determine the formation of a rigid and heavily cross-linked polymer network around the inert filler particles (1). Physical and mechanical properties of the composite resins are in direct relation to the extent of this polymerization reaction, to adequate curing or degree (effectiveness) of cure (2). Higher solubility of the resin, poor mechanical propertie, adhesion failure, and pulp toxicity due to residual monomers are some effects of inadequate light curing process (3-10).

Bulk-fill composites were released on the market with the purpose to decrease the time needed for composite application by decreasing the number of composite layers which have to be individually polymerized. These materials also avoid some problems associated with layering technique like contamination during the procedure, bonding failure, and difficulty in placement in small cavities with limited

access (11). They also decrease the polymerization shrinkage and the resulting stress (12) when using the same exposure time and light intensity as conventional composite resins as a result of decreasing the filler content, altering the filler matrix composition to increase material translucency and changing the photoinitiator system (13). One disadvantage of these materials is the incomplete polymerization in deep cavities (14) probably due to the reduction of light intensity while light passes through composite layer of 4–5 mm. The degree of cure at the bottom of composite surface as a result of scattering, absorption, and reflection seems to be similar or even lower when comparing to conventional composite resin light cured in layers of 2 mm thickness (13).

The first bulk-fill composite resin (Surefil SDR® flow, Dentsply Caulk) appeared on the market in 2010 and the producer recommanded its application in increments up to 4 mm. The main indication for use of this composite resin was as a base in class I and class II due to its consistency which is similar to that of a flowable resin. Unfortunately, mechanical strength and wear resistance were far from reaching the values of a conventional composite resin, so an additional layer of 2 mm of conventional composite resin is needed in the area of occlusal loading. Chemical composition of bulk-fill composites is much similar to the conventional ones, but classic Bowen monomer (Bis-GMA:2,2-bis[4-(2-hydroxy-3-methacryloxyprpoxy) phenyl] propane) has been modified by monomers having lower viscosity (Burgess J, 2010; Peutzfeldt A., 1997) or other monomers like Bis-GMA, UDMA, TEGDMA, and EBPDMA have been added in the matrix (15). The patented resin of Surefil SDR® flow based on urethane dimethacrylate monomer ensures a decreased polymerization stress due to a greate molecule flexibility (16). The amount of fillers in bulk-fill flowable resins is lower than in conventional microhybrid and nanohybrid compositeresins, but very similar to the conventional flowable composites by volume and higher by weight. Increased filler size (20μm) and lower filler loading determine the increased curing depth of these materials by improving the light penetration and by reducing the difference in refractive index between resin matrix and fillers (17, 18).

The degree of composite cure may be evaluated by different methods: infrared spectroscopy, laser Raman spectroscopy visual, scrapping and microhardness testing (19). Incremental microhardness testing and hardness ratio value, give by the bottom hardness value divided by top hardness value multiplied by 100 is considered an effective method to evaluate the degree of curing (2). To consider that the composite resin has been adequately cured, a hardness ratio above 80% has been suggested as the lowest threshold accepted value (20).

The aim of the study was to evaluate the influence of some characteristic of the light curing unit (polymeriyation mode and the distance between light curing tip to the surface of composite resin) on the efficiency of a bulk-fill base resin polymerization.

MATERIAL AND METHOD

SDR® Plus bulk fill flowable base composite shades U (universal), A2 and A3 was used in the study. Fiftyfive samples from each shade were prepared by

injection the flowable resin in cylindrical plastic molds with the intern diameter of 5 mm. Three types of plastic molds according to their hight were used: 6 mm, 8 mm, and 10 mm. The manufacturers indicate to use this material as a base, so except the 4 mm preserved for the flowable composite layer, other 2 mm were preserved in the mold for the covering layer of conventional composite resin and dditional 0 mm, 2 mm and 4 mm were preserved as the distance from the tip of light curing unit to the surface of the restoration. The whole distance between the tip of the lamp and the surface of the material was 2mm, 4mm and 6mm (by adding 2 mm of covering layer with 0mm, 2mm, and 4 mm from the tip of the lamp and the surface of final restoration). On the plastic molds a line was marked at 4 mm distance from the bottom in order to establish the hight of of the future flowable composite resin samples. The molds were placed in contact with a Mylar matrix strips on a glass slab in order to obtain a flat surface. SDR Pus was injected from the compules directly into the molds until the marked line, in this way one bulk layer of composite resin of 4 mm was obtained. The samples were light cured using a LED light unit having a intensity of 1.000 mW/cm²–1.200 mW/cm² and a wavelength of 385–515 nm (LED G, Woodpecker) in three different modes: normal, staggared and inclined. The tip of the light curing unit was placed on top of the mold and composite resin was light cure for 20 seconds for U shade and 40 seconds for A2 and A3 shades, according to the indications provided by the producer.

Top and bottom surfaces of the samples were ground flat, than all de samples were submersed in distilled water for 24 hours. After that the samples were distributed in groups (I-III according to the distance from the tip of the light curing unit to the surface of the composite resin) and subgroups (1-3 according to the polymerization mode), as they are presented in Table 1.

Table 1. Distribution of the samples in groups and subgroups

SDR shade	Distance of polymerization	Number of samples		
		Subgroup 1- NP	Subgroup 2-SP	Subgroup 3-IP
U	2 mm-Group I	5	5	5
	4 mm-Group II	5	5	5
	6 mm-Group III	5	5	5
A2	2 mm-Group I	5	5	5
	4 mm-Group II	5	5	5
	6 mm-Group III	5	5	5
A3	2 mm-Group I	5	5	5
	4 mm-Group II	5	5	5
	6 mm-Group III	5	5	5

NP-Normal polymerization mode; SP- Staggared polymerization mode; IP-Inclined

polymerization mode

Top and bottom surface Vickers hardness was determined using a digital microhardness tester CV 400 DAT (Namicon). Micro-indentations were made using the diamond squared had of the machine having an angle of 136 degree and a diameter of 2.5 mm. A fixed load of 50 g was applied. To accept an indentation, it should be sharp on diagonal lines, to be uniform, without irregularities in the testing area. The length of the axes was registered and the Vickers hardness Number (VHN) was calculated according to the formula: $D = 1854,4 \cdot F/d^2$ (N/mm^2) (D is the Vickers hardness number, F is the testing force, d is the indentation length of the diagonal). The VHN for each surface was reported as a mean of three readings. The hardness ratio of each specimen was also calculated.

RESULTS

Examples of micro-indentation aspects on some samples in tested groups are presented in Fig. 1-3. For SRD Plus flowable composite resin shade U samples, in group I subgroup 1top VHN values varied between 48.71 and 44.47, in subgroup 2 between 47.01and 42.85, in subgroup 3 between 42.35 and 37.91 (Table 2). Bottom microhardness varied in group I subgroup 1 between 44.42 and 40.08, in subgroup 2 between 41.64 and 38.12, in subgroup 3 between 38.83 and 34.69 (Table 3).In group II subgroup 1the VHN values varied between 45.51 and 40.41, in subgroup 2 between 43.35and 39.01, in subgroup 3 between 40.28 and 36.02 (Table 2). Bottom microhardness varied in group II subgroup 1 between 41.21 and 36.71, in subgroup 2 between 39.32 and 36.06, in subgroup 3 between 37.01 and 32.71 (Table 3). In group III subgroup 1the VHN values varied between 42.01 and 38.25, in subgroup 2 between 42.45 and 37.79, in subgroup 3

between 38.94 and 34.6 (Table 2). Bottom microhardness varied in group III subgroup 1 between 39.19 and 34.35, in subgroup 2 between 36.15 and 32.37, in subgroup 3 between 34.11 and 30.19 (Table 3).

For SRD Plus flowable composite resin shade A2 samples, in group I subgroup 1 the VHN values varied between 45.13 and 40.95, in subgroup 2 between 41.34 and 38.oo, in subgroup 3 between 36.19 and 32,29 (Table 2). Bottom microhardness varied in group I subgroup 1 between 40.39 and 36.65, in subgroup 2 between 37.71and 33.63, in subgroup 3 between 35.42 and 31.92 (Table 3). In group II subgroup 1 the VHN values varied between 43.33 and 38.63, in subgroup 2 between 39.18 and 36.46, in subgroup 3 between 35.35and 31.59 (Table 2). Bottom microhardness varied in group II subgroup 1 between 38.33 and 35.43, in subgroup 2 between 36.28 and 31.96, and in subgroup 3 between 31.57 and 28.67 (Table 3).

For SRD Plus flowable composite resin shade A3 samples, in group I subgroup 1 the VHN values varied between 44.08 and 40.85, in subgroup 2 between 40.89 and 36.79, in subgroup 3 between 37.21 and 33.35 (Table 2). Bottom microhardness varied in group I subgroup 1 between 37.94 and 34.62, in subgroup 2 between 33.80 and 30.62, and in subgroup 3 between 31.77 and 28.67 (Table 3). In group II subgroup 1 the VHN values varied between 41.43 and 38.11, in subgroup 2 between 38.92 and 34.6, in subgroup 3 between 33.7 and 30.82 (Table 2). Bottom microhardness varied in group II subgroup 1 between 34.15 and 31.17, in subgroup 2 between 31.76 and 28.82, and in subgroup 3 between 31.62 and 28.66 (Table 3). In group III subgroup 1the VHN values varied between 40.93 and 37.49, in subgroup 2 between 37.34 and 32.9, in subgroup 3 between 33.7 and 30.58

(Table 2). Bottom microhardness varied in group III subgroup 1 between 33.63 and 30.99, in subgroup 2 between 31.39 and 28.93, and in subgroup 3 between 30.56 and 27.94 (Table 3).
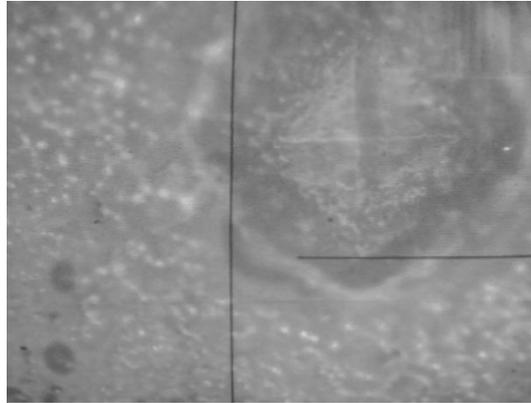

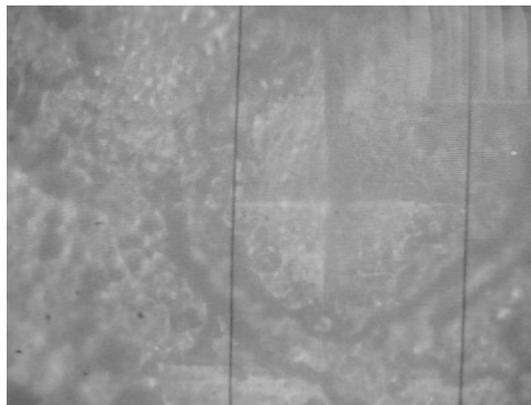**Figure1. Aspect of an indentation of SRD Plus shade A3 group I subgroup 1**

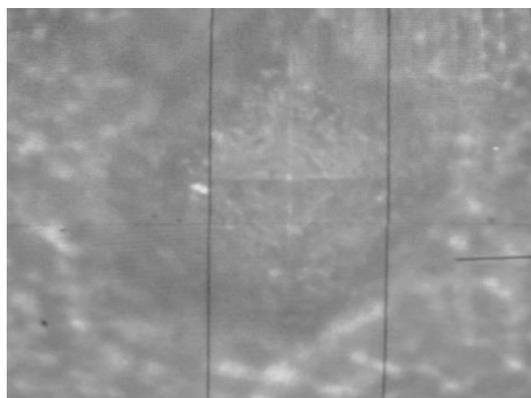**Figure2. Aspect of an indentation of SRD Plus shade A3 group II subgroup 2**

Figure 3. Aspect of an indentation of SRD Plus shade A3 group III subgroup 3

The highest top VHN mean value was obtained din group I subgroup 1 for U shade and the lowest in group III subgroup 3 for A3 shade (Table 2). In the same subgroup the VHN mean values decreased from group I to group III. In the same group of samples, a decrease of mean VHN values was recorded from the subgroup 1 to subgroup 3. Also, the mean VHN valued decreased when comparing U shades to A2 and A3 shades, respectively (Fig. 4).

Table 2. Top microhardness in groups and subgroups

| SDR shade	Distance of polymerization	Top microhardness-mean VHN±SD		
		Subgroup 1- NP	Subgroup 2-SP	Subgroup 3-IP
U	2 mm-Group I	46.59±2.12	44.93±2.08	40.13±2.22
	4 mm-Group II	42.96±2.55	41.18±2.17	38.15±2.13
	6 mm-Group III	40.13±1.88	40.12±2.33	36.77±2.17
A2	2 mm-Group I	43.04±2.09	39.67±1.67	34.24±1.95
	4 mm-Group II	40.98±2.35	37.87±1.41	33.47±1.88
	6 mm-Group III	39.95±1.40	36.35±1.59	33.12±1.56
A3	2 mm-Group I	42.44±1.59	38.84±2.05	35.28±1.93
	4 mm-Group II	39.77±1.66	36.76±2.16	32.26±1.44
	6 mm-Group III	39.21±1.72	35.12±2.22	32.14±1.56
NP-Normal polymerization mode; SP-Staggared polymerization mode; IP-Inclined polymerization mode; VHN-Vickers hardness number; SD-standard deviation				
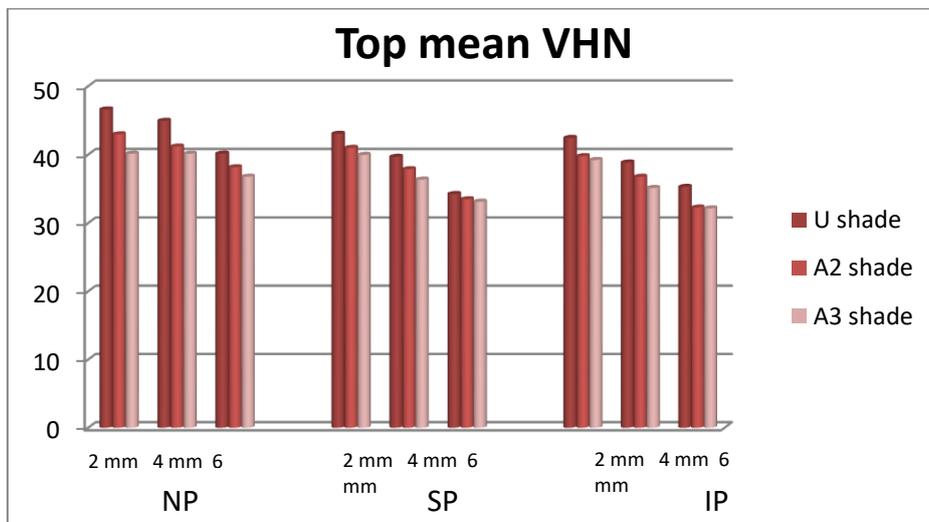
Figure 4. Mean top hardness values variation in groups and subgroups

The highest bottom VHN mean value was obtained din group I subgroup 1 for U shade and the lowest in group III subgroup 3 for A3 shade (Table 3). In the same subgroup the VHN mean values decreased from group I to group III. In the same group of samples, a decrease of mean VHN values was recorded from the subgroup 1 to subgroup 3. Also, the mean VHN valued decreased when comparing U shades to A2 and A3 shades, respectively (Fig. 5).

Table 3. Bottom microhardness in groups and subgroups

| SDR shade	Distance of polymerization	Top microhardness-mean VHN±SD		
		Subgroup 1- NP	Subgroup 2-SP	Subgroup 3-IP
U	2 mm-Group I	42.25±2.20	39.88±1.76	36.76±2.07
	4 mm-Group II	38.96±2.25	37.69±1.63	34.84±2.17
	6 mm-Group III	36.76±2.43	34.26±1.05	32.15±1.96
A2	2 mm-Group I	38.52±1.87	35.67±2.04	33.67±1.75
	4 mm-Group II	36.88±2.35	34.12±2.16	30.12±1.88
	6 mm-Group III	35.12±1.73	33.59±1.76	30.87±1.45
A3	2 mm-Group I	36.28±1.66	32.21±1.59	30.22±1.55
	4 mm-Group II	32.66±1.49	30.32±1.44	30.14±1.48
	6 mm-Group III	32.31±1.32	30.16±1.23	29.25±1.31
NP-Normal polymerization mode; SP-Staggared polymerization mode; IP-Inclined polymerization mode; VHN-Vickers hardness number; SD-standard deviation				
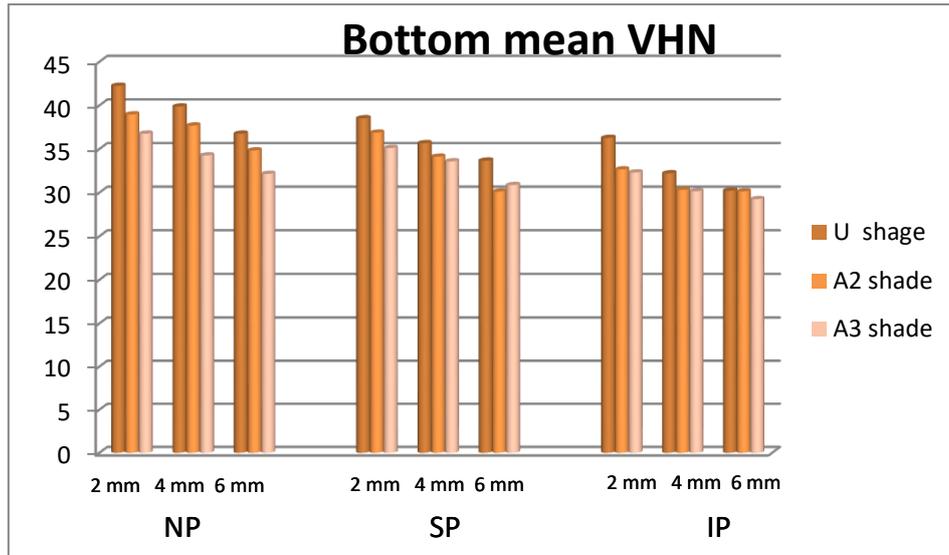
Figure 5. Mean bottom hardness values variation in groups and subgroups

A deacrease of VHN values was registered in evry group and subgroup when comparing top and bottom hardness (Tables 2 and 3). For tested material the hardness ratio was above 80%, so the composite resin was adequately cured, irrespective the distance of the curing unit tip to the material surface or the polymerization mode (Table 4).

Table 4. Mean hardness ratio in groups and subgroups

SDR shade	Distance of polymerization	Mean VHN top/bottom ratio ±SD		
		Subgroup 1- NP	Subgroup 2-SP	Subgroup 3-IP
U	2 mm-Group I	90±0.9	88±0.10	91±0.10
	4 mm-Group II	90±0.9	91±0.8	91±0.8
	6 mm-Group III	91±0.8	85±0.10	87±0.9
A2	2 mm-Group I	89±0.9	89±0.9	98±0.10
	4 mm-Group II	89±0.9	90±0.8	89±0.8
	6 mm-Group III	87±0.8	92±0.9	93±0.8
A3	2 mm-Group I	85±0.9	82±0.5	85±0.9
	4 mm-Group II	82±0.7	82±0.5	93±0.8
	6 mm-Group III	82±0.9	85±0.8	91±0.9
NP-Normal polymerization mode; SP-Staggared polymerization mode; IP-Inclined polymerization mode; VHN-Vickers hardness number; SD-standard deviation				

DISCUSSION

One of the major advantages when using bulk-fill composite resins is the possibility of using larger increments of material. To achieve this goal the manufacturers claim that the molecular formula of resin base has been modified by reducing or replacing Bis-GMA with a lower viscosity monomers or by using higher molecular weight monomers. In addition, to control the polymerization contraction they incorporated stress-relieving materials and changed the filler content. A variation of 1.8 to 3.3 % of the initial total volume of the material after light curing was reported in previous studies (21-27).

The curing depth of a light cured composite resin should not be less than 0.5 mm from the value established by the manufacturer, as it is stated in ISO 4049-2009 standard (28). A previous study evaluated the curing depth of ten different bulk-fill coposite resins and the results showed that the majority of these composites presented curing depth values equal or even greater than what required by the ISO for bulk-fill composites, but there were four materials which did not reach the values required by this standard (29). Other studies have also used the methodology indicated by the ISO specification, obtaining similar results (30). In both studies, of Tiba and Garcia, Surefil SDR reached the values specificated by the standard (average depth of cure: 5.01 mm) (29, 30). Also, other study recorded the curing depth values accepted by the standard for Surefil SDR and other three bulk-fill composite resins when being polymerized 20 seconds, but when measuring Vickers hardness the ISO standard overestimated the curing depth value (31).

Various results regarding mechanical properties of bulk-fill composite resins were recorded in the studies which evaluated the influence of increment thickness, time of light irradiation, and distance between the light curing lamp tip and the material surface (15, 32, 33). The greater curing depth reported for bulk-fill composite when comparing to conventionl ones can be explained by three mechanisms: a more efficient initiation systems (34), a greater translucency which allow a deeper light penetration by reducing light absorption by pigments, and decreased matrix/fill surface interface, which reduces light refraction (15).

There are various factors which can affect the polymerization process of composites such as the chemical composition of the resin, the type of filler, the increment thickness, the light curing sourse, the curing mode, the light curing time, the post-irradiation time, the cavity diameter and location, the distance between the light curing tip and material surface. Previous studies have demonstrated that increased filler/matrix ratio leads to progressive decrease of degree of conversion due to filler particles which act as obstacles in polymers chain propagation (35). Also, other parameters as light permeability of the fillers, type of the monomer, concentration of the initiator and co-initiator/inhibitor can affect the composite depth of cure and degree of conversion (36).

The results of some previos studies regarding the polymerization efficiency of bulk fill composite resins are of great variability. The results are mainly dependent on bulk fill composite resin brand and the time of light activation. Some studies

evaluated the effect of over or under exposure time (15, 37-40). Tarle et al concluded that the minimum curing times recommended by the manufacturers is not adequate for placement of high-viscosity bulk-fill materials (41). In vitro researches demonstrated that a radiant exposure of 12-24 J/cm^2 is needed to cure adequately a 2-mm layer of composites resin (42). Generally, the manufacturers do not make any specification regarding thelevel of radiant exposure needed to cure their products adequately and they indicate a fixed curing time for each composite increment and some may specify the minimum irradiance needed for light curing units. For example, for 4-mm increment of SDR, 10 s could be indicated using radiant exitance greater than 1000 mW/cm^2. Several studies showed that after 20 s of curing SDR bulk-fill composite yielded satisfactory polymerization in depth over 4 mm with the minimum irradiation times stated by the manufacturers (38, 39, 43, 44). Some authors recommanded extended curing time to achive better polymerization of bulk-fill composites in deep cavities (45).

The polymerization efficiency of SDR bulk-fill flowable composite at greater layer depth should probably be ascribed to its high translucency, which allow more transmission of light through the material. The study of Sarma A. et al. demonstrated that the measurement of transmitted light irradiance to a depth of 4 mm was higher for SDR bulk-fill flowable composite when comparing to other composite resins (46). Unfortunately, this high translucency represents a disadvantage for the aesthetics of the material. The producers devoped also bulk-fill composite resins having different shades, but the addition of various piments modified the time needed for a correct polymerization. In our study there was a

decreasing tendency of surface hardness variation for darker shades samples (A2 and A3) when comparing to universal shade (translucent) samples.

Different polymerization modes were tested in present study. In normal (standard or continuous) polymerization mode the light power remains constant at the same intensity from the beginning to the end of polymerization process and it is applied continuously in different time intervals: 10, 20, 30 or 40 seconds. In inclined (soft-start polymerization) a low light intensity is used initially and then it increases gradually. This allows a slow rate of polymerization with formation of less free radicals and extends the time for the composite to flow on the surface. Viscoelastic phase of the composite is prolonged and shrinkage stress of the composite resin can be controlled by slowing the hardening rate (47). In staggared (oscillating polymerization) mode the lamp units apply a low intensity light followed immediately by a high intensity light. In our study inclined and normal polymerization modes determined higher values of surface hardnesswhen comparing to staggared mode.

CONCLUSIONS

Polymerization efficiency of SRD$^®$ Plus bulk-fill flowable base composite resin was not affected by the distance from the light sourse to the surface of the material and the light curing mode. Staggared polymerization mode of the light curing unit determines slightly low values of surface hardness when comparing to inclined and normal polymerization modes. Darker shades of bulf fill flowable base composite resin seems to present decreased surface hardness when comparing to ligther shades irrespective the distance from the light curing unit to the material surface and the mode of polymerization.

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