

Evaluation of the Degree of Conversion and Curing Depth for the Composite Resin

Amer A. Taqa^{1,*}, A.-H. A. Suliman², Aimer J. S. Al-Taee²

¹College of Dentistry, DBS. Department, University of Mosul, Mosul, Iraq ²College of Dentistry, Conservative Department, University of Mosul, Mosul, Iraq

Email address

amertaqa@hotmail.com (A. A. Taqa) *Corresponding author

To cite this article

Amer A. Taqa, A.-H. A. Suliman, Aimer J. S. Al-Taee. Evaluation of the Degree of Conversion and Curing Depth for the Composite Resin. *Advances in Biomedical Sciences*. Vol. 1, No. 1, 2016, pp. 1-9.

Received: May 29, 2016; Accepted: June 6, 2016; Published: June 24, 2016

Abstract

The aims of this study are to investigate the effect of light intensity, curing time and combined effect of light intensity and curing time on the degree of conversion, depth of cure and amount of residual monomer released from composite samples cured with different light curing units. Single light cured composite restorative material (Herculite XR) exposed to different types of light curing units (Optilight LD, ULTRA-LITE 200 E plus, Astralis-VIVADENT) at a different exposure times (20, 40 and 60 seconds). The degree of conversion of composite resin samples was investigated by Fourier-transform infrared spectroscopy. The depth of cure of composite resin samples was investigated by scraping technique using a digital caliper. While the amount of residual monomer released from cured composite samples was investigated through measuring the released residual monomer in doubled distilled water with (CECIL 2000) ultraviolet visible spectrometer. Analysis of variance (ANOVA), Post Hoc (T test) and Duncan multiple range test were used for statistical analysis. The result of this study showed that there is a significant difference in the degree of conversion, depth of cure and residual monomer of composite samples with increasing time, increasing intensity and the combined effect of increasing time and intensity except for the degree of conversion in which the combined effect of increasing time and intensity was non-significant. In addition to that, it was shown that the low intensity LED LCU (Optilight LD) exhibit a non-significant result in degree of conversion from those of high intensity (LED and QTH) LCUs at extended period of curing time.

Keywords

Degree of Conversion, Light Intensity, Curing Time, Residual Monomer, Composite

1. Introduction

The use of light to polymerize composite resin has increased dramatically in the last few years [1] [2] The polymerization requires sufficient intensity of light and suitable wave length that match the absorption curve of camphorquinon (a visible-light sensitive dikitone photoinitiator responsible for initiating free-radical polymerization) present traditionally in adhesive and resin composite systems [2] [3]. Camphorquinone (CQ) absorbs energy in the visible-light region of 400-500 nanometers with peak at 468 nanometers. Photons associated with this frequency range will be absorbed by CQ rising it from the ground state to an excited activated state. This activated state of CQ will enter a

reaction with amine co-initiator (present in composite resin) leading to the formation of amino alkyl free radical which initiate polymerization reaction [4]. Manufactures have turned their attention to the light source used to convert composite materials from monomers to polymers [5].

Researches have investigated the relationships among curing source intensity, exposure duration, and tip- to- tooth curing distance to achieve optimal resin cure [6], [7].

Although halogen bulb based light curing units (LCUs) are most commonly used to cure dental composites, this technology has inherited many drawbacks. Halogen bulb have a limited effective life time of around 50 hours, the bulb reflector and filter degrade with time due to high temperature produced leading to reduction in light output. The result is a reduction of the LCUs effectiveness to cure dental composites, the clinical implication of this for dentist is a negative effect on the physical properties of composites with an increased risk of premature failure of restorations [8] [9].

To overcome the problems inherent to halogen LCUs, solid-state light emitting diode (LED) has been proposed for curing light -activated dental materials. Rather than a hot filament as used in halogen lamps, LED uses junction of doped semiconductors (P-n junction) for the generation of blue light. No filters are required in LED LCUs because the spectral output of Gallium-Nitride blue LED falls conveniently within the absorption spectrum of the CQ photoinitiator (400-500 nm) present in light-cured dental composite, furthermore LEDs have an expected life time of several thousand hours without significant degradation of light intensity over time [8]-[10].

The depth of cure of light-curing resin composites is an important property because insufficient cure of the deep part of a composite restoration implies stiffness, and insufficient bonding to the tooth structure [11]. Also, it is desirable for a dental resin composite to convert all of it is monomer to polymer during the polymerization reaction. Adequate polymerization is a crucial factor in obtaining optimal physical properties and clinical performance of resin composite restorative materials [12].

The degree of conversion defined as the percentage of reacted C=C bond (aliphatic) to the unreacted C=C bond (aromatic), this degree of conversion will affect many properties including mechanical properties, solubility, color change and biocompatibility of the resin composite [13], [14]. Thus it has been postulated that the degree of conversion plays an important role in determining the ultimate success of restoration [12], [14].

The aims of the Study: First: To evaluate the degree of conversion of composite resin in respect to different curing units. Second: to evaluate the depth of cure of composite resin in respect to different curing units. Third to evaluate the correlation between the light intensity, emitted by different curing units, monitored by radiometer, and the degree of conversion and depth of cure of composite resin Fourth: to evaluate the correlation between the exposure time on the degree of conversion and depth of cure of composite resin. Fifth: to measure the amount of residual monomer released from composite resin in respect to different curing units. And sixth: to evaluate the correlation between the exposure time and light intensity emitted by different curing units, and the amount of residual monomer released from composite resin.

2. Materials and Method

Tested Material: The material used in this study was the visible light-curing dental restorative herculite XR resin composite (Syborn/sds Kerr, USA). The LED sample preparation for degree of conversion: For the evaluation of the ratio of conversion of the reacted C=C (aliphatic) to the unreacted C=C (aromatic), composite samples were prepared by condensing the composite material into a plastic mold (1mm depth and 8mm in diameter) placed on glass slap. After

placing the composite resin into the mold, it was covered by celluloid strip and cured by Optilight LED curing unit for 20 second at intensity of 130 mW/cm². After curing the material was removed from the plastic mold and crushed and grinned manually by piston and mortar into a powder. Then the powder was mixed with potassium bromide and compressed into a disc shape by the Bruker press at a load of 10 tons.

The degree of conversion of the irradiated samples was measured by Fourier transform infrared spectroscopy (FTIR) (Bruker 27, TENSOR, Germany). The degree of conversion on the tested samples was calculated by the two frequency techniques using the net peak transmittance areas of C=C stretching vibration at 1638 cm⁻¹as analytical frequency and the aromatic C=C stretching vibration at 1608 cm⁻¹ as reference frequency [16]-[18].

2.1. The QTH Samples Preparation for Degree of Conversion

The composite samples were prepared in the same method like the LED samples but they were cured with a QTH light curing unit (Astralis, VIVADENT, and Austria).

2.2. The Uncured Sample Preparation for Degree of Conversion

The composite samples were prepared by mixing the composite material with CCl_4 , for dilution, and then compressing the composite material between two glass slaps supplied by the manufacturers of the spectrometer device and the measurement were made for the aliphatic C=C reacted bond and the C=C aromatic unreacted bond in the same manner for the cured composite [12[16], [17], [7], [18].

The LED sample preparation for the depth of cure: Curing depth of the materials was determined by means of a scraping technique described by International Organization of Standardization (ISO 4049, 1989).

The material of composite resin was filled into a stainless steel mold with a cylindrical opening 4mm in diameter by 10mm in height Figures 1. The surface was covered by a celluloid strip from the top and by glass slap from the bottom. The mold was exposed from the top to the curing light (LED) for 30 seconds; immediately after curing, the composite material was removed from the mold, and uncured material was scraped off with an alcohol-treated gauze. The thickness of the cured material was measured at the central portion of the resulting cylinder using a digital caliper [19], [20].



Figure 1. Metal Mold (Fabricated Stainless Steel Split Mold).

2.3. The QTH Sample Preparation for Depth of Cure

The QTH sample preparation for depth of cure was prepared in the same way as in LED samples.

In this study, two different types of light curing units were used to evaluate the degree of conversion, depth of cure and amount of residual monomer released from composite samples. The first type was a LED LCU, which include two units (one with a low intensity output 140mW/Cm² and other with high intensity output 550mW/Cm^2), these unit respectively were: Light curing unit, LED type, (Optilight LD,GNATUS, Brazil) and Light curing unit, LED type, (ULTRA-LITE) 200E Plus. TAIWAN. The second type was a conventional QTH LCU, (Astralis, VIVADENT, and Austria). The intensity of this QTH LCU was controlled by a manual Intensity-changeable device, in order to produce two different intensities resemble those of the LED LCU, the intensity of the LCUs was measured by a Cromatest 7041 curing RADIOMETER(Germany), so the result was a two QTH LCU one with low intensity and another with high intensity. The final number of the fabricated samples tested in this study were three hundred sixty samples. These samples were divided into three main category:

I. For the determination of the degree of conversion a one hundred twenty samples were utilized these samples were divided into four groups one group for each light curing unit, and each group was divided into three sub groups representing different time of curing exposure (20, 40, and 60 Second).

II. For the determination of the depth of cure a on hundred twenty samples were utilized these samples were divided into four groups one group for each light curing unit, and each group was divided into three subgroups representing different time of curing exposure (20, 40, and 60 Second).

III. For determination of the amount of residual monomer released into the doubled-distilled water a one hundred twenty samples were utilized these samples were divided into four groups one group for each light curing unit, and each group was divided into three sub groups representing different time of curing exposure (20, 40, and 60 Second).

2.4. Measurements of Degree of Conversion

The degree of conversion of the irradiated samples were measured by Fourier transform infrared spectroscopy (FTIR) (Bruker 27, TENSOR, Germany).

2.4.1. Measurement of Depth of Cure

Immediately after curing of each specimen, it was removed from the split mold, and the uncured composite materials was scrapped off with an alcohol-treated gauze, the thickness of the cured materials was measured at the central portion of the resulting cylinder using a digital caliper (METR-ISO-GEW, China) [19], [21], [22].

2.4.2. Measurement of Residual Monomer

The time-dependant monomer concentration was followed by monitoring the amount of monomer present in supernatant medium using (CECIL2000) Ultraviolet-Visible spectrophotometer (λ =254) compared with pure monomer [23], [24]. A solution of 10 mg of pure methyl methacrylate (monomer) was diluted in 1 ml of alcohol to prepare a stock solution in a concentration of 10 mg/ml concentration of solution [25].

From this stock solution, a range of solution concentrations were prepared including: 5 mg/ml, 2.5 mg/ml, 1 mg/ml, and 0.5 mg/ml and 0.25 mg/ml concentration solutions. The ultra violet visible spectrophotometer (CECIL 2000) was used at λ =254 nm to measure the absorbency of these solutions. From the resultant measurements of these known concentrations of stock solutions, a linear calibration curve of methyl methacrylate concentration as a function of absorbency at λ =254 nm was obtained Figure 2. From this calibration curve, the concentrations of the released monomer from the tested samples, were obtained by incidence the absorbency values obtained from the spectrophotometer on the calibration curve to obtain the concentration values.

Regression Plot

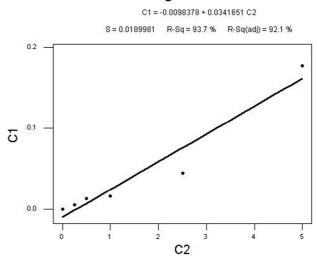


Figure 2. Liner Calibration Curve.

C1=Absorbance (nm)

C2=Concentration (mg/ml)

3. Results

The number of samples, mean and standard deviation of samples cured with different curing time (20,40 and 60 seconds) and different curing intensities (low and high), are arranged respectively in Table 1.

	TIME(Sec)	INTENSTY (mW/Cm ²)	Mean	Std. Deviation	Ν
DEPTHLED*	20	Low intensity	3.4910	7.637E-02	10
		High intensity	5.1940	.1173	10
	40	Low intensity	4.1940	.1394	10
		High intensity	6.0900	.2276	10
	60	Low intensity	4.4730	7.987E-02	10
		High intensity	6.5060	8.822E-02	10
DEPTHHAL	20	Low intensity	3.5480	8.244E-02	10
		High intensity	4.9530	.1231	10
	40	Low intensity	4.2800	7.242E-02	10
		High intensity	5.8620	5.453E-02	10
	60	Low intensity	4.7870	2.406E-02	10
		High intensity	6.3250	8.935E-02	10
CONLED	20	Low intensity	53.5156%	1.7037	10
		High intensity	56.6483%	9.1432	10
	40	Low intensity	69.3267%	5.7873	10
		High intensity	75.3488%	6.0619	10
	60	Low intensity	73.7223%	7.8567	10
		High intensity	78.3611%	7.0075	10
CONHAL	20	Low intensity	52.1315%	7.2404	10
		High intensity	52.5178%	3.4305	10
	40	Low intensity	54.3939%	11.0689	10
		High intensity	62.1280%	3.2245	10
	60	Low intensity	59.6747%	1.5011	10
		High intensity	67.9913%	5.4057	10
RMLED*	20	Low intensity	2.3179	6.301E-02	10
		High intensity	1.6301	7.690E-02	10
	40	Low intensity	1.6137	9.601E-02	10
		High intensity	1.3781	7.581E-02	10
	60	Low intensity	1.3123	6.618E-02	10
		High intensity	1.2328	3.466E-02	10
RMHAL	20	Low intensity	2.5837	5.077E-02	10
		High intensity	1.8110	.1894	10
	40	Low intensity	2.0000	7.442E-02	10
		High intensity	1.5644	8.669E-02	10
	60	Low intensity	1.7808	7.098E-02	10
	00	High intensity	1.2876	6.611E-02	10

Table 1. Mean, Standard Deviation and Number of Samples Utilized in This Study.

*DEPTHLED= Depth of cure for samples cured with LED LCU (mm).

DEPTHHAL= Depth of cure for samples cured with QTH LCU (mm).

CONLED= Degree of conversion for samples cured with LED LCU (%).

CONHAL= Degree of conversion for samples cured with QTH LCU (%).

RMLED= Amount of residual monomer released form samples cured with LED LCU (mg/ml).

RMHAL= Amount of residual monomer released form samples cured with QTH LCU (mg/ml).

Statistical analysis of two way ANOVA test shows the effect of time, intensity and the effect of time and intensity (combined together) on the degree of conversion, depth of cure and the residual monomer released from composite samples. It was obvious from this analysis that there was a significant difference (p<0.05) in degree of conversion (CON), depth of cure (DEPTH) and residual monomer (RM) of composite samples regarding increase in time, increase in intensity and increase in time and intensity (combined together), with the exception that there was no significant difference in the degree of conversion of composite resin regarding increase in time and intensity (combined together) beyond 20 seconds Table 2.

Table 2. Tow Way ANOVA Test Showing the Effect of Time, Intensity and Time and Intensity (Combined Effect).

Source	Dependent Variable	Type III Sum of Squares	df	Mean Square	F	Sig.
CorrectedModel	DEPTH LED	66.978(a)	5	13.396	765.605	.000
	DEPTH HAL	51.629(b)	5	10.326	1599.582	.000
	CON LED	5343.266(c)	5	1068.653	23.963	.000
	CON HAL	1970.519(d)	5	394.104	10.345	.000
	RM LED	7.811(e)	5	1.562	308.007	.000
	RM HAL	9.640(f)	5	1.928	189.894	.000
Intercept	DEPTH LED	1494.805	1	1494.805	85433.673	.000
-	DEPTH HAL	1475.600	1	1475.600	228584.877	.000
	CON LED	275976.998	1	275976.998	6188.460	.000

Source	Dependent Variable	Type III Sum of Squares	df	Mean Square	F	Sig.
	CON HAL	202812.439	1	202812.439	5323.513	.000
	RM LED	149.940	1	149.940	29563.367	.000
	RM HAL	202.678	1	202.678	19961.693	.000
TIME	DEPTH LED	13.837	2	6.919	395.421	.000
	DEPTH HAL	17.419	2	8.709	1349.148	.000
	CON LED	5005.274	2	2502.637	56.119	.000
	CON HAL	1324.865	2	662.433	17.388	.000
	RM LED	5.137	2	2.568	506.391	.000
	RM HAL	4.490	2	2.245	221.103	.000
INTENSTY	DEPTH LED	52.866	1	52.866	3021.473	.000
	DEPTH HAL	34.126	1	34.126	5286.458	.000
	CON LED	317.109	1	317.109	7.111	.010
	CON HAL	450.293	1	450.293	11.819	.001
	RM LED	1.676	1	1.676	330.484	.000
	RM HAL	4.825	1	4.825	475.252	.000
TIME*INTENSTY	DEPTH LED	.275	2	.137	7.855	.001
	DEPTH HAL	8.492E-02	2	4.246E-02	6.578	.003
	CON LED	20.884	2	10.442	.234	.792
	CON HAL	195.361	2	97.680	2.564	.086
	RM LED	.998	2	.499	98.386	.000
	RM HAL	.325	2	.163	16.005	.000
Error	DEPTH LED	.945	54	1.750E-02		
	DEPTH HAL	.349	54	6.455E-03		
	CON LED	2408.153	54	44.595		
	CON HAL	2057.264	54	38.097		
	RM LED	.274	54	5.072E-03		
	RM HAL	.548	54	1.015E-02		
Total	DEPTH LED	1562.727	60			
	DEPTH HAL	1527.578	60			
	CON LED	283728.417	60			
	CON HAL	206840.221	60			
	RM LED	158.024	60			
	RM HAL	212.866	60			
CorrectedTotal	DEPTH LED	67.922	59			
	DEPTH HAL	51.978	59			
	CON LED	7751.419	59			
	CON HAL	4027.782	59			
	RM LED	8.085	59			
	RM HAL	10.189	59			

Table 3. Multi-comparisons Test (Effect of Curing Time).

Dependent Variable	TIME(Sec)	TIME	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence	Interval
						Lower Bound	Upper Bound
DEPTH LED	20	40	7995(*)	4.183E-02	.000	9048	6942
		60	-1.1470(*)	4.183E-02	.000	-1.2523	-1.0417
	40	20	.7995(*)	4.183E-02	.000	.6942	.9048
		60	3475(*)	4.183E-02	.000	4528	2422
	60	20	1.1470(*)	4.183E-02	.000	1.0417	1.2523
		40	.3475(*)	4.183E-02	.000	.2422	.4528
DEPTHHAL	20	40	8205(*)	2.541E-02	.000	8845	7565
		60	-1.3055(*)	2.541E-02	.000	-1.3695	-1.2415
	40	20	.8205(*)	2.541E-02	.000	.7565	.8845
		60	4850(*)	2.541E-02	.000	5490	4210
	60	20	1.3055(*)	2.541E-02	.000	1.2415	1.3695
		40	.4850(*)	2.541E-02	.000	.4210	.5490
CONLED	20	40	-17.2558(*)	2.1118	.000	-22.5716	-11.9400
		60	-20.9597(*)	2.1118	.000	-26.2755	-15.6439
	40	20	17.2558(*)	2.1118	.000	11.9400	22.5716
		60	-3.7039	2.1118	.224	-9.0197	1.6119
	60	20	20.9597(*)	2.1118	.000	15.6439	26.2755
		40	3.7039	2.1118	.224	-1.6119	9.0197
HAL	20	40	-5.9363(*)	1.9519	.014	-10.8496	-1.0230
		60	-11.5084(*)	1.9519	.000	-16.4216	-6.5951
	40	20	5.9363(*)	1.9519	.014	1.0230	10.8496

Dependent Variable	TIME(Sec)	TIME	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval		
						Lower Bound	Upper Bound	
		60	-5.5721(*)	1.9519	.022	-10.4853	6588	
	60	20	11.5084(*)	1.9519	.000	6.5951	16.4216	
		40	5.5721(*)	1.9519	.022	.6588	10.4853	
RMLED	20	40	.4781(*)	2.252E-02	.000	.4214	.5348	
		60	.7014(*)	2.252E-02	.000	.6448	.7581	
	40	20	4781(*)	2.252E-02	.000	5348	4214	
		60	.2233(*)	2.252E-02	.000	.1666	.2800	
	60	20	7014(*)	2.252E-02	.000	7581	6448	
		40	2233(*)	2.252E-02	.000	2800	1666	
RMHAL	20	40	.4151(*)	3.186E-02	.000	.3349	.4953	
		60	.6631(*)	3.186E-02	.000	.5829	.7433	
	40	20	4151(*)	3.186E-02	.000	4953	3349	
		60	.2480(*)	3.186E-02	.000	.1678	.3282	
	60	20	6631(*)	3.186E-02	.000	7433	5829	
		40	2480(*)	3.186E-02	.000	3282	1678	

 Table 4. Paired Samples Test (T test) Effect of Intensity on Degree of Conversion and Depth of Cure.

		Mean	Std. Deviation	Std. Error Mean	95% Confidence Interval of the Difference				
Intensity/Time		Mean	St. Deviation	Ctd. Error mean	Lower	Upper	t	Df	Sig.
Pair 1depth of cure	LowLED 20 HighLED 20	-1.7030	.1306	4.129E-02	-1.7964	-1.6096	-41.249	9	.000
Pair 2 depth of cure	LowLED 40 HighLED 40	-1.8960	.2744	8.676E-02	-2.0923	-1.6997	-21.854	9	.000
Pair 3 depth of cure	LowLED 60 HighLED 60	1.7790	.2154	6.811E-02	1.6249	1.9331	26.120	9	.000
Pair 4 depth of cure	Low HAl 20 High HAl 20	-1.3330	.2394	7.569E-02	-1.5042	-1.1618	-17.611	9	.000
Pair 5 depth of cure	Low HAl 40 High HAl 40	-1.5850	.1073	3.394E-02	-1.6618	-1.5082	-46.705	9	.000
Pair 6 depth of cure	Low HAl 60 High HAl 60	-1.5980	.2114	6.685E-02	-1.7492	-1.4468	-23.906	9	.000
Pair 7 conversion	Low LED 20 High LED 20	.6877	.1173	3.709E-02	.6038	.7716	18.543	9	.000
Pair 8 Conversion	Low LED 40 High LED 40	.2356	.1149	3.635E-02	.1534	.3179	6.482	9	.000
Pair 9 Conversion	Low LED 60 High LED 60	7.946E-02	8.113E-02	2.565E-02	2.142E-02	.1375	3.097	9	.013
Pair 10 Conversion	Low HAl 20 High HAl 20	.7810	.2012	6.364E-02	.6370	.9250	12.272	9	.000
Pair 11 Conversion	Low HAl 40 High HAl 40	.4300	.1494	4.726E-02	.3231	.5369	9.099	9	.000
Pair 12 Conversion	Low HAl 60 High HAl 60	.4497	.1916	6.058E-02	.3127	.5867	7.424	9	.000

Table 5. Paired Samples Test (T test) Effect of Intensity on Degree of Conversion and Depth of Cure.

Intensity/Time	Intensity/Time		erences				t	df	Sig. (2-tailed)
		Mean	Std. Deviation	Std. Error Mean	95% Confidence Interval of the Difference				
					Lower	Upper			
Pair 1 residual	LowLED 20 HighLED 20	.6877	.11728	.03709	.6038	.7716	18.543	9	.000
Pair 2 residual	LowLED 40 HighLED 40	.2356	.11495	.03635	.1534	.3179	6.482	9	.000
Pair 3 residual	LowLED 60 HighLED 60	.0795	.08113	.02565	.0214	.1375	3.097	9	.013
Pair 4 residual	Low HAL 20 High HAL 20	.7727	.18349	.05802	.6414	.9039	13.317	9	.000
Pair 5 residual	Low HAL 40 High HAL 40	.4357	.13137	.04154	.3417	.5296	10.487	9	.000
Pair 6 residual	Low HAL 60 High HAL 60	.4932	.06458	.02042	.4470	.5394	24.150	9	.000

3.1. Effect of Curing Time

Multi comparisons Test and Post Hoc Test (regarding the effect of curing time) shows that there was a significant difference (P<0.05) in degree of conversion, depth of cure and residual monomer released in all groups as the time of cure increased, except for the degree of conversion in high intensity LED LCU in which the change in degree of conversion was not significant beyond 20 seconds curing time Table 3.

3.2. Effect of Time on Residual Monomer of Samples Cured With QTH

The Paired Samples test (T test) (regarding the effect of intensity), shows that there was a significant difference (P< 0.05) in degree of conversion, depth of cure and residual monomer released in all the groups as the intensity of curing light increased. Duncan's multiple range for all groups of samples (different times and different intensities) showed Tables 4, 5:

Or, First; Degree Of Conversion: The HLED 60, H LED 40 and L LED 60 gave the highest degree of conversion followed by L LED 40 and H HAL 60, followed by H HAL 40, L HAL 60 and H LED 20. Second; Followed by LHAL40and LLED 20, followed by HHAL 20 and LHAL20. Third; For Residual Monomer: The HLED60, HHAL60 and LLED60 gave the least amount of residual monomer followed receptively by HLED40 Sec, HHAL40 Sec, LLED40, HLED20, HHAL20, LHAL40, LLED20, LHAL20.

4. Discussion

The FTIR has proved to be a powerful technique for the analysis of degree of monomer conversion in dental composite, and in turn to give an idea about the characteristics of the resultant cured samples regarding their physical properties[18][3], [16].

In addition, the scraping test of depth of cure[21], [19], [22] (Tanoue *et al.*, 1998; Tanoue *et al.*, 1999; Pradhan *et al.*, 2002), and measurement of the amount of residual monomer released in double distilled water[25], [23], [24], have been found to be beneficial measures of the characteristics of the cured composite samples.

4.1. Effect of Intensity

The result of this study showed that there was a significant difference in the degree of conversion of composite resin, depth of cure and the amount of residual monomer (the degree of conversion and depth of cure measures were increased with increasing light intensity while the residual monomer measures were reduced with increasing light intensity) as the intensity of curing light increased [14], [16], [26], [8], [5], [27].

A plausible explanation for such effect of the light intensity on the prepared composite samples could be attributed to the important effect of light intensity regarding composite resin polymerization, as the intensity represents the number of photons delivered to the composite resin per unit of time. These delivered photons are considered responsible for the process of photopolymerization as they activate the sensitive photoinitiator complex present in the composite resin, which intern give rise to the free radical polymerization cascade [4].

A remarkable event in this study, that both the low and high intensities LED LCUs produced a better results regarding the degree of conversion, depth of cure and the amount of residual monomer than their antagonizing QTH LCU.

A reasonable explanation for this event, is that the halogen lamp is more efficient in the red and infrared light and is only slightly energetic in the zone of CQ absorption of maximum at 470 nm. It is common knowledge that red light produces more heat than violet light. Unfortunately, this means that the temperature rises without significantly improving photopolymerization. Important advantage of blue LEDs is the possibility to choose the most efficient wave length of 470 nm, justifying the very narrow wave length preference of CQ. Any wavelengths below about 430nm and above 500nm are not utilized in the electron promotion of the ketone groups in CQ and therefore it can be said that CQ ignores these wave lengths. The unwanted wave lengths do produce additional heat, affecting the kinetics of the reaction and may thereby influence the reaction. Conventional light sources produce a white light, which is then filtered in an effort to remove the unwanted wave lengths [28], [7], [29].

4.2. Effect of Time

The result of this study showed that there was a significant difference in the degree of conversion, depth of cure and the amount of residual monomer (degree of conversion and depth of cure were increased while the residual monomer decreased), with increasing of time.

A reasonable explanation for this result is that time of irradiation at a given irradiance determines the total number of free radicals generated [27].

This result could be explained as the influence of power density on the extent of conversion is already illustrated through its relationship to the polymerization rate, the increase or continuous illumination time will lead to force the rate of free radical polymerization of acrylates and methacrylate's to follow a characteristic pattern throughout the course of reaction due to diffusion limitation on the reacting species. This pattern is manifested early in the reaction by a decrease in the radical termination rate and a concurrent increase in the radical concentration [26]. As a consequence, the rate of polymerization accelerates (auto acceleration) through a maximum despite a decreasing monomer concentration.

A remarkable event in this study regarding the effect of time, that when a low intensity LED LCU was used with

expanded curing time it produced non-significant results from the high intensity LCUS (LED and QTH) regarding the degree of conversion and the amount of residual monomer.

This event has been explained by a comparison between the two intensities LCUs, so in case of high intensity LCU, and after having passed through the maximum polymerization process as mentioned earlier, the rate begins to decrease due to continuation in monomer consumption. As the network develops further, the rate of radical propagation eventually becomes diffusion limited and the polymerization rate decelerates, often towards a limited conversion in the presence of unreacted monomer and a significant population of radicals. Decreasing power density will decrease the rate of polymerization and shift the maximum rate to longer times. Provided the irradiation time is not limited, conversion will continue through its diffusion limited maximum. If irradiation is terminated while propagation is chemically controlled, the final conversion will be reduced from its maximum. In the previous discussion, it was noted that in order to increase conversion with lower power density, longer exposure times are required.

Another explanation suggest that, while increased conversion was noted at the lowest intensities, this result occur using extremely long exposure times (even more than 60 seconds) and was explained from kinetic theory as being due to the dependency of kinetic chain length on intensity.

4.3. Effect of Time and Intensity

The result of this study showed that there was a nonsignificant change in the degree of conversion of composite resin with increasing both curing time and light intensity.

This is simply explained by that, each composite resin material has a maximum polymerization level in which the monomer is accelerated to reach this level either by increasing the light intensity, or by increasing the curing time. But the result of combined increase in time and intensity will only result in reaching this maximum polymerization rate more rapidly *i.e.* no additional polymerization beyond the maximum is obtained.

5. Conclusion

The conclusions of this study are as follows: first; there is a significant difference in the degree of conversion, depth of cure and residual monomer of composite resin with increasing curing intensity. Second; there is a significant difference in the degree of conversion, depth of cure and residual monomer of composite resin with increasing curing time. Third; there is a significant difference in the depth of cure and residual monomer, and a non-significant difference in degree of conversion with the combined effect of increasing time and intensity. Fourth; for an optimal polymerization, the emission spectrum of the curing source has to be closely matched to the absorption spectrum of the photoinitiators. Fifth; the curing of a material is to be conducted in such a way that internal stress can be reduced by giving the material time to flow before reaching the gel point. This is accomplished by using a low intensity light curing unit with increased curing time, and final sixth; the effectiveness of low intensity curing suggests a novel approach for resin composite restorations: the LED curing appliance which proved to exhibit a significant effect in curing dental resins.

References

- Narene A. V. K., Veniashok B., Subbiya A., Vivekanandhan P. and Sukumaran V. G.. Polymerisation Shrinkage in Resin Composites-A Review, Middle-East Journal of Scientific Research, 2014; 21 (1): 107-112, and references therein.
- [2] Rueggeberg, F. A, Caughman, W. F. and Curtis, J. W. Effect of light intensity and exposure duration on cure of resin composite. Oper Dent 1994.; 19, 26-35.
- [3] Tarle Z, Meniga A, Knezevic A, Sutalo J, Ristic M., Composite conversion and temperature rise using a conventional, plasma arc, and an experimental blue LED curing unit. 2002; J Oral Rehab; 29: 662-667.
- [4] Stansbury JW., Curing dental resins and composites by photopolymerization. 2002; J Esthet Dent; 12: 300-308.
- [5] Emami N, Soderholm K-JM. (2003): How light irradiance and curing time affect monomer conversion in light-cured resin composites. J Oral Sci; 111: 536-542.
- [6] Fernanda C. P. PIRES S., Brahim D. F., Luciana A. CASEMIRO, I. F. Roberti G. C., polymerization shrinkage stress of composites photoactivated by different light sources, Braz Dent J, 2009; 20(4): 319-324.
- [7] Rahiotis C, Kakaboura A, Loukidis M, Vougiouklakis G. Curing efficiency of various types of light-curing units. Eur J Oral Sci; 2004; 112: 89-94.
- [8] 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: 2955-2963.
- [9] Bennet AW, Watts DC., Performance of two blue lightemitting –diode dental light curing units with distance and irradiation time.2003; Dent Mater; 20: 72-79.
- [10] Stahl F, Ashworth SH, Jandt KD, Mills RW., Light-emitting diode (LED) polymerization of dental composites: flexural properties and polymerization potential. Biomaterials; 2000; 21: 1379-1385.
- [11] Munksgaard EC, Peutzfeldt A, Asmussen E., Elution of TEGDMA and BisGMA from a resin and a resin composite cured with halogen or plasma light. Eur J Oral Sci; 2000; 108: 341-345.
- [12] Yoon T. H.,Y. K. Lee, B. S. Lim and C. W. Kim., Degree of polymerization of resin composites by different light sources. J Oral Rehab; 2002; 29: 1165-1173.
- [13] Vaidyanathan J, Vaidyanathan TK. Interactive effects of resin composition and ambient temperature of light curing on the percentage conversion, molar heat of cure and hardness of dental composite resin. J Mater Sci: Mater Med, 1992. 3; 19-27.

- [14] Sobriho LC, Goes MF, Consani S, Sinhoreti MAC., Correlation between light intensity and exposure time on the hardness of composite resin. J Mater Sci: Mater in Med; 2000; 11: 361-364.
- [15] Peutzfeld A, Sahafi A, Asmussen E. Characterization of resin composites polymerized with plasma arc curing units. Dent Mater 2000; 16: 330-336.
- [16] Silikas N, Eliades G, Watts DC., Light intensity effects on resin composite degree of conversion and shrinkage strain. Dent Mater; 2000; 16: 292-296.
- [17] Knezevic A, Tarle Z, Meniga A, Sutalo J, Pichler G and Ristic M. Degree of conversion and temperature rise during polymerization of composite resin samples with blue diodes. J. Oral Rehabil, 2001 28; 586-591.
- [18] Uctasli S, Tezvergil A, Lassila LVJ, Vallittu PK, The degree of conversion of fiber-reinforced composites polymerized using different light-curing sources. Dent Mater; 2005; 21: 469-475.
- [19] Tanoue N, Matsumura H, Atsuta M. Curing depth of prosthetic composite materials polymerized with their property photocuring units. J Oral Rehab; 1999; 26: 594-599.
- [20] Rannjit D. Pradhan, Noureddine Melikechi, and Frederick Eichmiller. The effect of irradiation wavelength bandwidth and spot size on the scraping depth and temperature rise sin composite exposed to an argon laser or a conventional quartztungsten-halogen source. Dent Mater, 2002; 18: 221-226.
- [21] Tanoue N, Matsumura H, Atsuta M. Curing depth of four composite veneering materials polymerized with different laboratory photo-curing units. J Oral Rehab; 1998; 25: 348-352.

- [22] Pradhan RD, Melikechi N, Eichmiller F., The effect of irradiation wavelength bandwidth and spot size on the scraping depth and temperature rise in composite exposed to an argon laser or a conventional quartz-tungsten- halogen source. Dent Mater; 2002; 18: 221-226.
- [23] De Oliveira MB, LE ON LT, Del bel cury AA, Consani S., Influence of number and position of flasks in the monomer release, knoop hardness and porosity of a microwave-cured acrylic resin. J Oral Rehab; 2003; 30: 1104-1108.
- [24] Sadoon MM., Evaluation of repairing the acrylic denture base by using different materials, designs, and techniques. M. Sc thesis, Collage of Dentistry, University of Mosul.2004.
- [25] Azzarri MJ, Cortizo MS, Alessandrini JI., Effect of the curing condition on the properties of an acrylic denture base resin, microwave polymerized. J Dent; 2003; 31: 463-468.
- [26] Halvorson RH, Erickson RL, Davidson CL., Energy dependent polymerization of resin based composite. Dent Mater; 2002; 18: 463-469.
- [27] Musanjie L, Darvell BW., Polymerization of resin composite restorative materials: exposure reciprocity. Dent Mater; 2003; 19: 531-541.
- [28] Uhl A, Mills RW, Jandt KD., Photoinitiator dependent composite depth of cure and knoop hardness with halogen and LED light curing units. Biomaterials; 2003; 24: 1787-1795.
- [29] Uhl A, Sigusch BW, Jandt KD., Second generation LEDs for the polymerization of oral biomaterials. Dent Mater; 2004; 20: 80-87.