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Assessing the Impact of Titanium Dioxide Nanotube Incorporation on the Coefficient of Thermal Expansion and Degree of Conversion of 3D-Printed Denture Base Resin

Zaid Basil Ali [(1) *]
Ihab Nabeel Safi [(2)]

[(1,2)] Department of Prosthodontics, College of Dentistry, University of Baghdad, Baghdad, Iraq.

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***Corresponding Author:**
Email:
Zaid.Ali2301m@codental.uobaghdad.edu.iq
Master's student, Department of Prosthodontics, College of Dentistry, University of Baghdad, Baghdad, Iraq.

Abstract

Hot or cold food and drink consumption causes temperature fluctuations in the oral environment. A dental prosthesis fitted in such environments might thermally expand or contract in response to heat fluctuations. A significant difference in thermal expansion characteristics between various denture parts (denture bases, artificial teeth, etc.) might result in the development of interfacial stress, which leads to a shortened denture shelf-life. This study aims to assess the effects of incorporation of titanium dioxide (TiO_2) nanotubes (NTs) into 3D-printed denture base resin on the coefficient of thermal expansion (CTE) and the degree of conversion (DC%). TiO_2NTs with a dimeter of 30-70nm and length of 2-4μm were incorporated into the 3D-denture base liquid resin at 0.0wt% (control), 1.0 and 2.0wt% groups. Total 30 digitally printed samples for coefficient of thermal expansion with (n=10) samples were tested for each subgroup and were analyzed by a Thermomechanical analyzer. Attenuated Total Reflectance -Fourier transform infrared spectroscopy was performed to analyze degree of conversion utilizing total 26 samples with (n=6) for each of the photo-polymerized 3D-printed groups samples (control, 1.0wt.% and 2.0wt.% TiO_2NT composite) and uncured 3D-printable liquid resin samples as baseline. The results showed that increasing TiO_2NT concentration enhance the coefficient of thermal expansion and no significant differences for degree of conversion. The clinically significant is that: The improvement in the coefficient of thermal expansion of 3D-printed resin owing to the addition of TiO_2NTs could diminish marginal weakening at the border between different denture parts, resulting in extended denture shelf-life.

Introduction:

PMMA is the most commonly used material for complete dentures (1). However, a major issue with traditional heat curing is that the shrinkage of the resin during polymerization interferes with the fit between the denture-bearing tissues and the PMMA denture base (2).

Digital denture production may be achieved using subtractive milling (SM) (3) or, recently, 3D additive printing (4-8). Denture prosthesis is most often printed using stereolithography (SLA) (9-13), digital light processing (DLP) (11-15) and lately, liquid crystal display (LCD) (5, 11). Building structures using 3D additive printing methods requires assembling them layer by layer (16). So, the underlying oral tissues are better fitted by digitally printed complete denture when compared to the conventional heat curing process (6, 17). Also, the number of visits needed until the final denture is delivered is also reduced (6), and can produce specimens with less material waste and lower costs (5, 18). However, as compared to traditional heat-cured methacrylate resins, their mechanical and thermal properties are still lacking (16, 19, 20).

To overcome the challenge of poor quality of 3D printable resin, investigators examined different techniques to strengthen the 3D printed resin by adding nanosized material (NM) as filler to improve the features of these dental materials (16). The properties of the 3D-printable resin composite material are influenced by the parameters of the polymer matrix and the type, size, shape, and concentration of the NMs used(15, 16, 21). The increased surface-to-volume ratio gives these NMs unique physical and chemical properties that set them apart from their bulkier equivalents (22).

Polymeric nanocomposites (PNCs) are made of a polymer matrix and nanoscale filler (23-27). A minimum of one dimension at the nanoscale(1–100 nm) is required of all nanomaterials (NMs) (28). The TiO_2 nanotubes (NT) are known as nanomaterials that have a diameter less than 100 nm and are made from titanium oxide, which is a naturally occurring element. It has great hydrophilicity, biocompatibility, chemical and thermal stability, low toxicity, and an outstanding refractive index and strength (16).

The degree of conversion of monomers (DC%) refers to the functional monomer groups proportion (e.g., acrylate groups) that take on polymerization to form polymers during the process of polymerization (29, 30). The DC% is affected by the monomers' dimensions, functional units, temperature, and the initiator mechanisms (31, 32). In the case of photo-induced polymer, the light source parameter including; wavelength, light intensity, curing time are also affecting the DC% (32). A limited DC% is due to the marked drop in radial chain-ends mobility (33-35) of suspending methacrylate and other functional monomers at raised cross-linked densities (35), ranging from 40–70% (36). Literature data specify that DC levels never reach 100% (33).

The oral environment faces various challenges during normal function. Food and drink can cause temperature fluctuations; according to one study, in vivo temperature changes can range from 0.8°C to 60°C after consuming hot or cold fluids. Dentures may expand or contract in response to these temperature variations. One factor that contributes to marginal deterioration is interfacial strain, which can result from significant differences in the thermal expansion properties of the various denture components (37).

The majority of nanocomposite research has concentrated on analyzing mechanical and physical features, with limited studies addressing the effect on thermal properties of 3D-printed dental (18, 38). Furthermore, prior research has limited the application of TiO_2 NPs to improving composites' mechanical, physical, and antimicrobial properties (39). To the author's knowledge, no research has investigated how the thermal expansion coefficient and degree of conversion are affected by the incorporation of TiO_2 NT filler. Consequently, this research set out to determine how adding TiO_2 NTs to 3D-printed denture base materials affected their CTE and DC%. According to the null hypothesis, adding TiO_2 NTs to 3D-printed denture base material does not

significantly alter the coefficient of thermal expansion or the degree of conversion characteristic.

Materials and Methods

Study design

The3D printable denture base resin liquid (DentaBase /Asiga, Australia) was utilized for specimen production via 3D- printing. 3D printed nanocomposites were created by incorporating titanium dioxide nanotubes (TiO_2 NT) (Hongwu, China) with a dimeter of 30-70 nm and a length of 2-4 μm. The nanotubes were used in concentrations of 1.0 and 2 weight percent (wt%). By comparing these findings to those of a control group that got no TiO_2 NT at all. The study was performed as part of MSc thesis at the Department of Prosthodontics, Collage of dentistry, University of Baghdad. And the tests done in Ministry of Science and Technology from November, 2024 to march, 2025.

Specimen preparation

The computer-aided design (3D Builder /Microsoft) program was applied for digitally building samples as shown in Figure (1-A). The samples were digitally generated with adequate dimensions in compliance with the testing specifications for the thermomechanical analyzer (TMA) device which used for coefficient of thermal expansion(CTE) analysis, these samples comprised of cylinder with (a diameter =5 mm and length=20 mm) as stated in ISO 11359-2 (40) as shown in Figure (1-A). The degree of conversion was analyzed by Attenuated Total Reflectance -Fourier transform infrared spectroscopy (ATR-FTIR), with 24 samples including six samples for each photopolymerized 3D-printed (control, 1.0% and 2.0wt%) groups, with addition six samples of uncured 3D printable liquid resin before polymerization which used as bassline.

Titanium dioxide nanotube incorporation

A combination of 99.9% ethanol (Honeywell, Germany) and titanium nanotubes (TiO_2 NT) (Hongwu, China) was subsequently made at a ratio of 3 mL of alcohol /1 g of nanotube. Then, the mixing ratio of the groups' added nanotube /3D printable liquid resin (control, 1.0 wt.% and 2.0 wt.%) was 0g /100 g, 1g /99g and 2g /98g, respectively. The next step was to put the mixture through three minutes of ultrasonic treatment using an MSE soniprep 150 from the United Kingdom. A shaded yellowish-brown glass bottle blended the 3D printing liquid resin with a TiO_2 NT solution. The lid was placed on top to shield the mixture from outside light. For 30 minutes at 60°C, the denture base resin and TiO_2 NT suspension were mixed using the Alfa HS-860 magnetic stirrer. During this time, the cover was slightly opened to allow the alcohol to evaporate. After that, the mixture was mixed at ambient temperature for 8 hours, while having the cover closed to avoid light from entering as shown in Figure (1-B).

Printing of 3-dimensional sample blocks

A digital light processing (DLP) 3D-printer from Asiga, Australia, was used to produce the samples as shown in Figure (1-C). When the printer's vat was filled to the brim with the liquid polymer mixture, the lid was secured to prevent the blend from exposure to light. The samples were printed with a horizontal orientation, parallel to the platform base, at an angle of 0 degrees.

The DLP printer algorithm divides the digital model into 50 μm thin horizontal layers to print the samples. As shown in Figure (1-D), the samples were exposed after the vat's contents were emptied of liquid after solidification.

Cleaning, drying, and curing

A sharp blade delicately removed the specimens from the 3D printer platform. The samples were then subjected to two rounds of 99.9% isopropyl alcohol ultrasonic cleaning (Clean I, Ackureta, Taiwan) for approximately three minutes

each. This procedure is performed to eliminate any remaining uncured resin. The samples were dried to a suitable degree before being put through a light curing process of polymerization in a UV box (Ackureta, Taiwan) with 385 nm LEDs at 65 watts of power for 30 minutes. The samples attached to the platform and supports were polished and fine-tuned using an acrylic bur and a lathe polishing device.

Thermomechanical analysis (TMA) procedure

A TMA device was used for coefficient of thermal expansion (CTE or α) tests. The test specimen was placed in the center of the specimen holder, the tapered tip probe was placed over the center of the specimen, and the temperature increase rate was 5º K/min.

Attenuated Total Reflectance -Fourier transform infrared spectroscopy (ATR-FTIR) test for degree of conversion (DC%) analysis

The FTIR spectrum parameters were mid-infrared (MIR), wavelength =4000-400 cm^{-1} with 2cm^{-1} resolution. The 3D printable liquid resin (before polymerization) was scanned as a baseline record ,and compared to the groups after polymeization (The control , 1.0 % wt and 2.0 % wt TiO_2 NT nanocomposite).Six specimens (n=6) for each of the four groups were tested. During polymerization, the C=C double bond in the monomer breaks down and is converted to a single bond in the subsequent polymer chain. To analyse the DC as a percentage, the absorbance spectra of the C=C functional peak in the cured polymer were measured and compared to the corresponding peaks in the 3D printable liquid resin (before polymerization).The values observed are regulated against a constant standard bond to account for variations in the specimens quantity. The C=O bond (34) with a peak frequency of 1712 is selected as the constant reference based on the tested material and test results obtained .The C=C stretching vibration peak is observed at 1649 cm^{-1} for the control, 1.0%, and 2.0% Specimens post-polymerization and curing. So, it was utilized as a standard for comparison between groups. The DC was

$$DC(\%) = \left(1 - \frac{\left(\frac{T_{C=C}}{T_{C=O}}\right)peak\ hights\ after\ polymerization}{\left(\frac{T_{C=C}}{T_{C=O}}\right)peak\ hights\ befor\ polymerization}\right) \times 100 \quad (41, 42)$$

calculated using the following equation:

Statistical analysis

The data analysis was done using Prism 8.4 (GraphPad Software, USA) to analyze the data. The descriptive analysis includes using a bar to show the results, including the mean values and standard deviations. Repeated-measures ANOVA, which, with post hoc Tukey's HSD tests, discloses various outcomes for the same sample when testing for different temperatures. Differences are considered nonsignificant when the p-value is more than 0.05, significant when the p-value is less than 0.05, and extremely significant when the p-value is less than 0.013.

Results

Coefficient of thermal expansion (CTE or α)

The findings of the coefficient of thermal expansion (α) are illustrated in Figure (2), after comparing the control and nanocomposite groups at various temperatures (30 °C, 40 °C, 50 °C, 60 °C, 70 °C). The CTE of the same group rises with increasing temperature, establishing a direct correlation between CTE and temperature. For the control group, with a mean value of (72.127×10^{-6}/K) at 30 ºC, and reaching a maximum mean value of (114.14×10^{-6}/K) at 70 ºC. Secondly, when comparing multiple groups at the similar temperature, CTE diminishes as the TiO_2 NT filler content increases, indicating an inverse correlation between CTE and the TiO_2 NT filler content. The mean value is 66.755×10^{-6}/K at 30ºC for the 1.0% TiO_2 NT composite, while the minimum mean value is 64.055×10^{-6}/K at 30ºC for the 2.0 wt % TiO_2 NT composite group, in contrast to the control group, exhibits a mean value of 72.127×10^{-6}/K at 30ºC. Significant differences were found between the control group and the other nanocomposite groups. The mean value, standard deviation, standard error,

maximum, minimum, repeated measure ANOVA of the coefficient of thermal expansion test results are displayed in Table (1). post-hoc Tuckey's test for Multiple pairwise of CTE between Groups are shown in Table (2).

Degree of conversion

Figure (3) shows Degree of conversion (DC%) test results with a mean value of 50.4167% for the control group, and 50.5167% and 50.7% for the 1.0% and 2.0% nanocomposite groups, respectively. No significant differences were measured between groups. The mean value, standard deviation, standard error, maximum, minimum and one-way ANOVA of the degree of conversion test results are displayed in Table (3).

Discussion

The clinical significance of α lies in the fact that temperature fluctuations occur as a result of hot or cold food and drink consumption, which may cause deformation of dental prostheses over time (18, 37). A dental prosthesis fitted in such environments might thermally expand or contract in response to heat fluctuations. A significant difference in thermal expansion characteristics between different dental prosthetic parts (denture bases, artificial teeth, etc.) might result in the development of interfacial stress, which has been linked to marginal deterioration as one of the etiological factors (37). The reduction in α would reduce marginal deterioration at the interface between different denture parts during the denture service life as a result of thermal fluctuations in the oral environment (18, 37). Thus, it is essential to improve the thermal properties of 3D printed prostheses using a variety of methods, the most common of which is the incorporation of nanomaterials into the resin (20, 43). It was discovered that adding TiO_2 nanotubes to 3D printed denture base resin significantly changed its coefficient of thermal expansion(α). As the concentration of TiO_2 NTs increased to 1.0% and 2.0% wt, the α was improved. Therefore, it is necessary to reject the null hypothesis.

Printing technologies and parameters

Because digital light processing (DLP) projects the laser as a complete two-dimensional (2D) pattern onto a layer, allowing the union of layers, DLP photopolymerization is faster than stereolithography (SLA) (11). Due to their higher light intensity, DLP printers offer superior item manufacturing capabilities compared to liquid crystal display (LCD) printers (5, 44) and they are often cheaper than SLA printers, making them a better alternative for small dental clinics (15). Hence, a DLP printer was utilized for the present investigation.

The samples printed for this study have a 50 μm layer thickness. The reason for this is that samples with a 50 μm layer thickness showed better characteristics than those with a 100 μm thickness, which is thought to be caused by The 3D-printed object's defining characteristics are improved with decreasing layer thickness owing to better resin curing and fewer dimensional changes (45). together with reduced spaces between air voids (46).

The nanofiller's precipitation and aggregation were reduced by conducting printing on a horizontal plane (0^{o} orientation) to shorten printing time (47).

Nanomaterial selection and filler content

This study used one-dimensional (1D) nanomaterials—specifically, titanium dioxide nanotubes (TiO_2 NTs)—as fillers. The x and y dimensions of these NTs are within the nanoscale range, but the z-dimension is larger than 100 nm (considered the nanoscale threshold). The end product is elongated materials with a needle-like structure, which increases the accessible surface area (48-51).

According to several studies, the best results are usually achieved with small concentrations of nanomaterials (19, 27, 52). Since most of 3D printing resins can handle 1.0 and 2.0 wt.% filler without major printing issues, our study restricted the concentrations of TiO_2 NT to those ranges. This is due to: First, the likelihood of printing errors grows in direct

proportion to the filler concentration (53). Second, it makes the resin liquid thicker, which might lead to inadequate recoating between layers and, ultimately, print failure (53). Third, because the dispersion from the fillers could impact interlayer curing, using a lot of fillers might lower UV light penetration and curing depth (53). Fourth, mechanical and thermal performance might be negatively impacted if the porosity is amplified during printing due to the formation of voids between consecutive layers of materials caused by an increase in NT concentration (46).

Coefficient of thermal expansion (CTE or α)

The coefficient of thermal expansion can be described as the proportion of the change in the dimension of the sample to its initial dimension when the temperature varies, represented as $\alpha = \Delta L/L^{\circ}\ \Delta T$ (54). A CTE mismatch between adjacent layers or parts in systems can result in structural degradation, such as interfacial delamination, cracking (55). The filler can either enhance or reduce the coefficient of thermal expansion, depending on the particular type or amount employed (54).

The coefficient of thermal expansion test results indicate two major findings when comparing groups at various temperatures (30ºC, 40ºC, 50ºC, 60ºC, and 70ºC). Initially, when the same group is evaluated at varying temperatures, α increases with rising temperature. This phenomenon arises from an increase in the kinetic energy of the atoms and molecules inside the structure, resulting in more intense vibrations and motions of its components. Rising temperatures result in broadening the distances between atoms and molecular chains. Moreover, when the temperature nears or surpasses the polymer's glass transition temperature, CTE often increases dramatically, resulting in considerable thermomechanical stresses inside the structure (56). Secondly, when several groups are examined at the same temperatures, CTE lowers as the concentration of TiO_2 nanofiller increases, which agrees with the findings of Safi. This may result from enhanced interfacial contacts between the nanofillers and the resin matrix, which restricts the mobility of chains and macromolecules (57). The decrease in CTE could minimize marginal degradation at the junction of various denture components during the denture's lifespan due to temperature fluctuations in the oral environment caused by the ingestion of hot and cold beverages and meals.

Mhaibes et al; noticed that TiO_2 NTs incorporation at 1.0 wt% and 1.5 wt% to 3D printed denture base material boosted its mechanical and physical characteristics, leading to a possible enhancement in clinical practice (19). Anti et al. address that the mechanical properties of TiO_2 NP-PMMA nanocomposite improved when TiO_2 NP concentration was around 2.5 wt.% and deteriorated when the amount of TiO_2 NTs exceeded 3.0 wt.% (58). When comparing these reports with the current results, it was also found that the addition of 2.0 wt.% TiO_2 NT to 3D printed resin could produce optimum mechanical, physical and thermal properties for denture base.

Degree of conversion analysis

The clinical relevance of DC% is that the mechanical properties of 3D printed resin including α are greatly affected by DC% (1, 59).

FTIR is a prevalent technique for assessing the degree of double-bond conversion, as it can identify the stretching vibrations of carbon-carbon double bonds participating in polymerization. During polymerization, the C=C double bond is opened and converted to a single bond in the polymer chain. (1, 31, 41).

The Degree of conversion (DC%) mean value test results are shown in Figure (3). The low degree of conversion of the control group (50.4167%) can be explained by: In 3D printed denture base resins, complete conversion of aliphatic carbon-carbon double bonds is generally not attained. The unconverted double bonds present in the resin result from two primary sources: unreacted monomers and pendant double bonds located at the ends of polymer chains (60).Also, during

polymerization, monomers experience a sequence of chemical events to produce polymer chains. Initially, monomers exist in a liquid form, allowing for unrestricted movement and interaction with neighboring monomers. The elevated mobility facilitates the effective deliver of reactive species, enhancing polymerization and leading to the progressive rise of the degree of conversion until the gel point, at which polymer chains become interlinked where the mobility of the monomers diminishes markedly. The reduction in monomer mobility following the gel point hinders their diffusion and reaction with other monomers, which may affect the DC (31). This reduction in monomer conversion increase in methacrylate resin at increased cross-linked level (35). Besides, the large Specimens size printed in this research (disc with 40 mm diameter and 15 mm thickness, and cylinder with 20mm length and 5mm diameter) which was done according to ISO standardization for polymer would limit penetration of curing light also long printing time result in sedimentation and affect the homogeneity of final layers which alter the composition which in return affect the degree of conversion.

Due to the photocatalytic properties (photoinduced process) of TiO_2 NT (61), adding TiO_2 NT accelerate photopolymerization. However, in this research, the addition was in low percentage (at 1.0 wt% and 2.0 wt%) the result was (50.5167% and 50.7%) respectively with alternation were limited to (<0.5%) compared to control group (50.4167%), and the addition of TiO_2 NT at (1.0 and 2.0)wt% have no significant effect on degree of conversion, this agree with finding of Ibraheem et al study (62).

Clinical significance, limitations and future work

Recent research suggests that 3D-printed materials may someday replace traditional materials in the fabrication of denture bases. Integrating TiO_2 NTs into 3D printed denture base resin offers practical benefit. As there is a reduction in the coefficient of thermal expansion. As there is a reduction in the coefficient of thermal expansion, it suggests that the interface among distinct denture components would experience lower marginal deterioration, resulting in increased clinical performance and longer shelf-life when in function. It was not possible to directly compare the optimal percentages of TiO2 NTs found in this work with those from earlier investigations on the use of TiO2 NTs in 3D-printed dental resin, as there is a lack of research on the thermal properties of 3D-printed acrylic.

Conclusions

A DLP printer using 1.0 wt.% and 2.0 wt.% TiO_2 NTs successfully produced nanocomposites resin for denture bases, within the constraints of this investigation. Both the 1.0 wt.% and 2.0 wt.% TiO_2 NTs composite groups showed an improvement in thermal characteristics (coefficient of thermal expansion), with the improvement being proportional to the quantity of nanotube fillers added.

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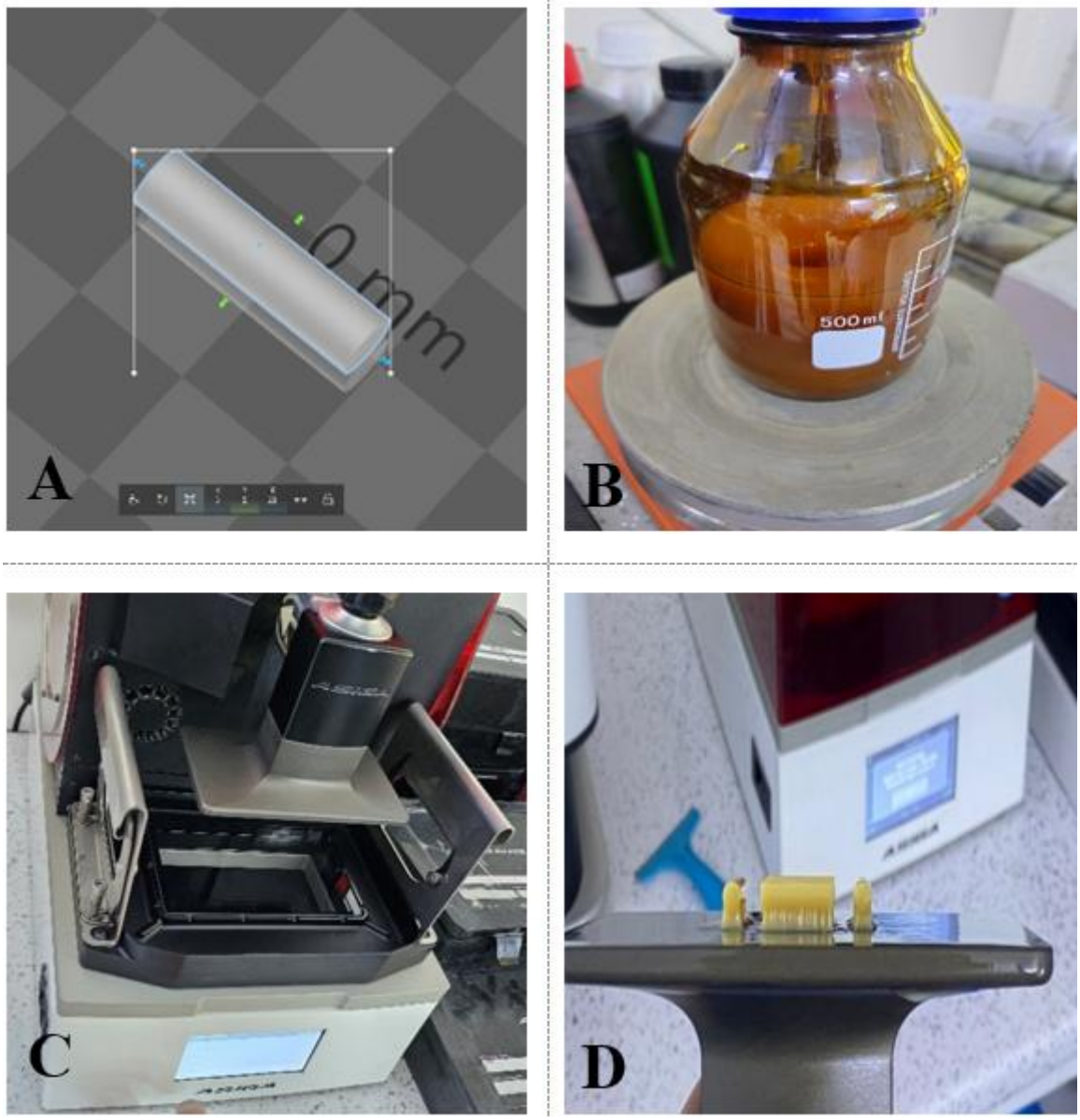

Figure (1): A. Dimensions for the coefficient of thermal expansion sample. B. 3D printed resin liquid stirring by magnetic stirrer. C. Asiga printer. D. samples on the platform after complete printing.

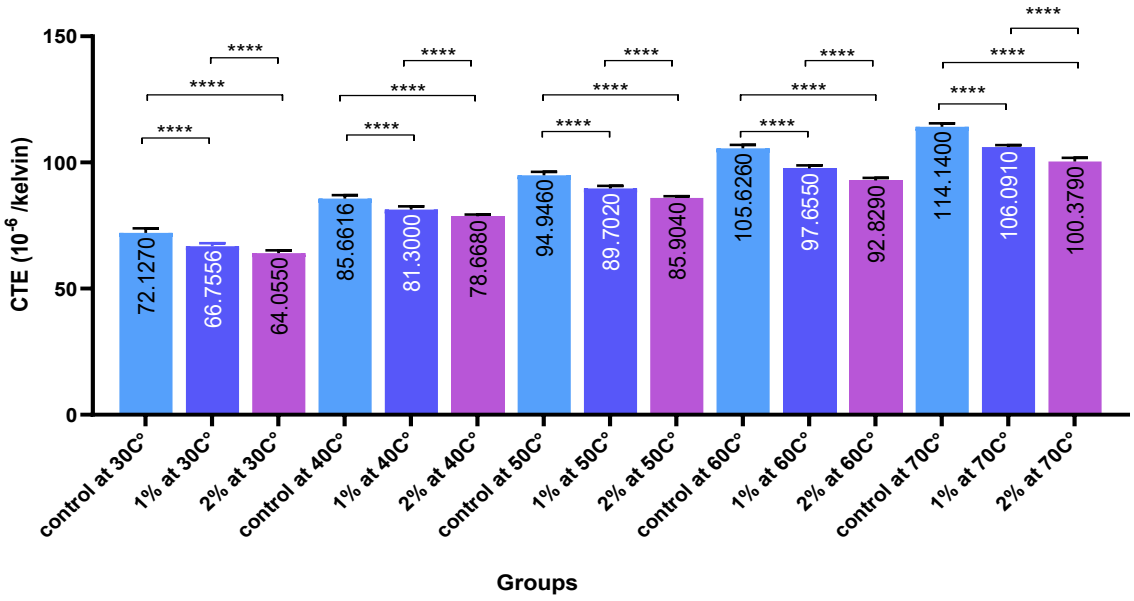

Figure (2): Coefficient of thermal expansion (α) × (10^{-6}/k) test results

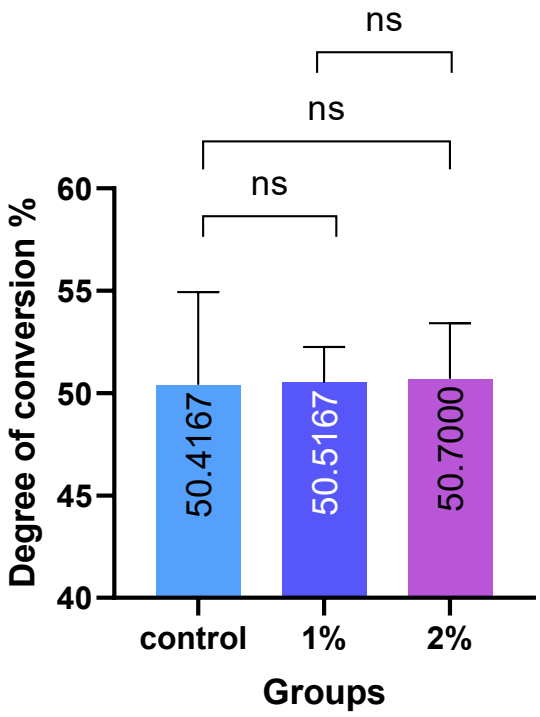

Figure (3): Degree of conversion (DC%) mean value.

Table (1): descriptive analysis, repeated ANOVA test for the coefficient of thermal expansion.

Descriptive test of CTE (10^{-6} /K) among groups and temperature.							Repeated ANOVA	
Groups		CTE30 ºC	CTE40 ºC	CTE50 ºC	CTE60 ºC	CTE70 ºC	F	P value
Control	Min.	70.090	83.140	92.730	103.890	111.400	1354.555	**0.000**
	Max.	74.970	87.270	96.760	107.740	116.300		
	Mean	72.127	85.6616	94.946	105.626	114.140		
	±SD	1.726	1.381	1.348	1.347	1.369		
	±SE	0.546	0.437	0.426	0.426	0.433		
1%	Min.	65.080	79.250	88.260	96.160	104.600	1149.943	**0.000**
	Max.	68.730	83.630	91.580	99.660	107.110		
	Mean	66.7556	81.300	89.702	97.655	106.091		
	±SD	1.264	1.288	1.036	1.182	0.783		
	±SE	0.400	0.407	0.328	0.374	0.248		
2%	Min.	62.840	77.320	84.770	91.270	97.000	969.834	
	Max.	65.980	79.690	86.840	94.600	102.280		**0.000**
	Mean	64.055	78.668	85.904	92.829	100.379		
	±SD	1.150	0.714	0.685	1.040	1.499		
	±SE	0.364	0.226	0.217	0.329	0.474		
Repeated NOVA		85.874	91.856	184.121	291.728	302.840		
		0.000	**0.000**	**0.000**	**0.000**	**0.000**		

Table (2): Post-hoc Tuckey's test Multiple pairwise of the coefficient of thermal expansion between Groups.

Post-hoc Tuckey's test Multiple pairwise of CTE between Groups						
Temp.	Groups		Mean Difference	p value	**95% CI**	
30	Control	1%	5.371	**0.000**	3.771	6.972
		2%	8.072	**0.000**	6.471	9.673
	1%	2%	2.701	**0.001**	1.100	4.301
40	Control	1%	4.362	**0.000**	3.031	5.692
		2%	6.994	**0.000**	5.663	8.324
	1%	2%	2.632	**0.000**	1.302	3.962
50	Control	1%	5.244	**0.000**	4.036	6.452
		2%	9.042	**0.000**	7.834	10.250
	1%	2%	3.798	**0.000**	2.590	5.006
60	Control	1%	7.971	**0.000**	6.605	9.337
		2%	12.797	**0.000**	11.431	14.163
	1%	2%	4.826	**0.000**	3.460	6.192
70	Control	1%	8.049	**0.000**	6.615	9.483
		2%	13.761	**0.000**	12.327	15.195
	1%	2%	5.712	**0.000**	4.278	7.146

Table (3): Descriptive statistics of degree of conversion (DC%) variables with ANOVA test.

Descriptive statistics of DC among groups.						ANOVA	
Groups	Mean	±SD	±SE	Minimum	Maximum	F	P value
Control	50.416	6.021	2.458	40.00	57.30	0.02007	0.9801 NS
1%	50.516	2.278	0.9300	47.20	53.70		
2%	50.70	3.549	1.449	45.00	54.70		

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