RESEARCH PAPER

In Vitro Evaluation of Ceramic-Amber Hardness Supported Zirconia

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Abstract: *Ceramics in dentistry have been mainly recommended from a cosmetic perspective. Yet, the hardness behaviour may limit the application in many cases. Although amber glass is used for medications and chemicals, no studies focus on using amber glass for dental purposes as an additive material. This study aims to investigate the behaviour of dark amber glass as a new additive material for dental ceramics. The amber glass powder was prepared using the ball mill technique. For the amber glass powder characteri-zation, the SEM/EDX, particle size, DSC, Ion release, and XRD analysis were tested compared to VITA Lumex® AC ceramic. In addition, the Vickers hardness test was applied for ceramic and ceramic amber, adding 0.01 g, 0.03 g, and 0.05 g of amber glass powder following the DIN EN ISO 6872/2019. Statistically, the ANOVA (post hoc-Tukey) test was used for hardness testing analysis at a significant P-value of (P≤ 0.05). The results show that the amber glass behaviour and composition elements seem similar to VITA ceramics. The addition of amber glass powder to ceramic shows an increase in the HV hardness of specimens. Overall, it was concluded that the amber glass powder could be a promising material for ceramics to use as an additive powder.*

Keywords: *Amber, Ceramic, Hardness, Zirconia, Powder, Dental, Characteristics.*

1. INTRODUCTION

Dental ceramics are widespread biomechanical materials for oral restorations. They are considered one of the most prevalent dental materials for partial and/or complete teeth restoration as inlays, onlays, crowns and overlays [1-3]. This could relate to acceptable mechanical properties such as abrasion, compression, bending, wear and fracture resistance [4]. In addition to chemical stability, there is both low thermal and electrical conductivity. However, regardless of the crystalline and amorphous ceramic structures and percentage, ceramics composed of SiO_2, Li_2O, K_2O, P_2O_5, Al_2O_3, ZrO_2, CeO_2, Na_2O, CaO, TiO_2, ZrO_2, Y_2O_3, and HFO_2 respectively [5, 6]. The brittleness behaviour may arise due to the presence of several flaws at small scales, including micro-cracks, microscopic notches, internal pores, grain misalignments, and impurities. Such imperfections might be increased during the manufacturing process of thermal gradients [7].

Material hardness is one of the mechanical properties that is frequently used as a parameter to evaluate the surface resistance of materials due to plastic deformation by penetration, indicating the ease of surface polishing or scratching [8]. The material hardness is an important parameter associated with many other ceramic properties and performance aspects. The ceramic hardness is typically affected by a function of both grain size and orientation, and chemical decomposition with porosity [9, 10].

Rice et al., who have reviewed the relationship between grain size and hardness of a variety of oxide ceramics, including MgO, BeO, Al_2O_3, $MgAl_2O_4$ and B_4C, as well as hydroxyapatite, have shown that an increase in grain size leads to a decrease in hardness [11]. In contrast, Wang et al (2016), the dependence of hardness on grain size does not always show a single trend that applies to every ceramic material, the hardness of some other ceramic materials such as TiB_2, SiC, TiC and Si_3N_4 is essentially independent of grain size. In addition, the average pore size increases with increasing temperature from 1100 to 1150°C. According to the general preparation for ceramics processing, using fine particles could result in higher densification and strength of sintered parts compared to large particles. It seems that the particle size affects the flowability and sinterability of the feedstock powder ceramic specimens [12]. While the Vickers hardness (HV), decreases rapidly as the porosity increases

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[13]. Nevertheless, research by Mei et al., shows that the Vickers hardness strongly depends on porosity and pore size. The hardness would decrease with increasing porosity and pore size [10]. Within the micrometric range, decreasing particle size results in chipping at lower loads due to increased contact pressure [14].

Due to weak interphases, ceramic-based dental composites are typically regarded as less intense and wear-resistant than monoliths. This generally results in quasi-plastic mechanical behaviour, which in turn decreases the resistance to chipping that results from the crack tip impeding propagation [14].

Recently, yttria-stabilized tetragonal zirconia polycrystal (Y-TZP) was offered to dental professionals. These materials must be manufactured using CAD/CAM (Computer-Aided Design/ Computer-Aided Manufacturing) techniques that have been tested in vivo for biocompatibility [15]. Partially stabilized zirconia has a higher fracture strength and structural reliability than glass ceramics when formed into a prosthetic framework. Nevertheless, due to zirconia's limited translucency, all zirconia frames are suggested to be veneered with glass ceramics or porcelain for aesthetic reasons [16, 17].

Glass is an amorphous material with high silica (SiO_2) content that is included in most dental materials, like glass ceramics and glass ionomer cement [18-22]. The glass may launch in different thermal and optical characteristics like flint, blue, green, light and dark amber [23, 24]. The amber glass is one of these available glass materials. They have many adsorption UV light bands that help in packaging purposes for beverages, medications, and chemicals, which are generally known for pharmaceutical packaging [24]. Dark amber bottles transmit substantially less than 2% of the total harmful wavelength light [25].

The amber glass contains many elements like iron oxides, carbon, sodium sulphate, calcium oxide, sodium oxide, and silica [26]. According to the Khorasani et al. study (2015), the higher micro silica particles to cement ratio from 2/1 to 2/2 a higher slump flow in diameter [27].

Yet, the World Health Organization (WHO) classified elements into either essential trace elements such as Cr, Cu, Zn, Se, Mo, and I; or the probably essential elements as Mn, Si, Ni, B, and V; or the potentially toxic elements represented by Al, F, Pb, Hg, Li, As, Sn, and Cd [28, 29].

The most controversial element concentrations for human safety are copper (Cu) and aluminium (Al). The copper (Cu) element is considered one of the essential trace elements accepted in small amounts of 5-20 (µg/g) and a total content of 100-150 mg in average adult humans due to carbohydrate metabolism and the functioning of more than thirty enzymes. However, when the concentrations exceed 20 (µg/g), they are considered toxic [30, 31]. On the other hand, the total body burden of Al in healthy humans has been reported to be approximately 30-50 mg/kg body weight, and daily intake in food ranges between 3.4 to 9 mg/day [32]. Therefore, this study aims to investigate the characteristic behaviour of dark amber glass as an additive material for dental ceramics.

2. EXPERIMENTAL PROCEDURES

The pharmaceutical Amber glass (Omega 3, 200 ml, Hansal, Germany), dental ceramic VITA Lumex® AC (VITA Lumex® AC, Germany), and zirconia (Aconia, ST White, China) were used in this study.

2.1. Amber Glass Powder Preparations

The Amber glass powder was prepared using pharmaceutical dark brown amber glass bottles. Initially, the bottles were cleaned, and crushed to the cullet manually using a hammer. The cullet was ground using a rotary horizontal ceramic ball-mill machine for 18h (110 rpm, CAPCO, UK). The ceramic balls were mostly circular with different sizes of 33-34, 26-28 and 22-25 mm. The resultant powder was sieved using 45 µm mesh grit at 10 Hz for 2hr (Retsh W. EML, Germany). A high-energy vertical mini-lap planetary ball mill (Henan, Nanbei, NXQM-4L, China) produced a fine particle size powder. Therefore, the planetary balls were made of chrome steel (Why ceramic balls haven't been used to avoid contamination?) with different sizes of 9.5, 8, 7 and 6mm. The powder/balls weight ratio was first 1:5 for 90 min at 200 rpm, then continued with a ratio of 1:10 at 200 rpm for seven h. To avoid heat generation, an intermittent pausing of 10 minutes was applied after each 30 minutes of the grinding procedure. Finally, the fine resultant powder of amber glass was dried in an electronic oven at 50°C for 30 min and then continued for complete dryness at 100°C for 15 min (BINDER Oven, USA).

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2.2. Material Characterization

For amber glass characterization, the resultant fine powder of amber glass material was analyzed alongside Vita dental ceramic for the element composition, morphology, particle size, and thermal behaviour. The SEM/EDX test (Quattro S-STEM/SEM, Czech Republic) was used to identify the composition and morphology of the tested material. The particle size was measured using a Malvern Mastersizer analyzer (Scirocco 2000, China).

The thermal behaviour was determined by Differential Scanning Calorimetry (DSC), and analysis was performed using **(**SDT Q600 V20.9 Build 20). The analyzed materials in this study are fine amber glass powder. Ceramic VITA Lumex® AC powder, VITA Lumex® AC with three different amber glass powder additive ratios of 0.01 g, 0.03 g, and 0.05 g using a precision balance (RADWAG AS 220/C/1).

In terms of Ion release, XRD analysis (How come XRD and not XRF?), and Vickers hardness test, the specimens were prepared using a silicone mould for study material of ceramic, ceramic-amber, and/or zirconia specimens with dimensions of 12×1.5 mm in diameter and thickness, respectively.

The ceramic and/or ceramic-amber specimens of 1.5 mm were prepared for the ion release test, and the Cu and Al released ions were measured after immersing the specimens in artificial saliva. The artificial saliva components and concentrations g/ml were followed [33]. These are of NaCl: 0.70%; KCl: 1.30%; $NaHCO_3$: 1.50%; $KH2PO_4$: 0.20%; Na_2HPO_4: 0.26%; and KSCN: 0.33%). All chemical materials were analytically graded (CDH, India) and the study was established in (pH= 7.3) for 7 days at room temperature with an Atomic absorption spectrometer and Graphite Furnace (AAS-GF) (AA-7000/ Japan).

On the other hand, for the XRD (ADX2700 Angstrom Advanced Inc, China) and Vickers hardness tests, the ceramic, ceramic-amber of 1mm thickness (How did you make the sample for making sure of a uniform thickness?) by using a silicone mlould with a zirconia substructure specimens of 0.5mm thickness was used, Fig 1. The Vickers hardness specimens were prepared following the DIN EN ISO 6872/2019 standardization for ceramic restorations [34-37]. The ceramic-amber powder/modelling liquid was mixed to form the dough for the specimen, layered over the zirconia substructure disc using the silicone mould for final reshaping. Each specimen was sintered at 800°C, then at 760°C using a porcelain furnace (Ivoclar Vivadent, Austria).

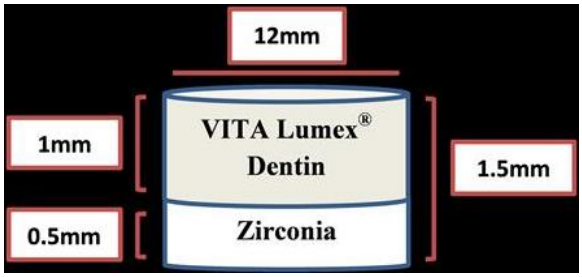

Fig. 1. Study specimens design

2.3. Vickers Hardness Test

The testing procedure for the Vickers hardness test was performed with Laryee hvs-5 (Manufacturing Limited, Beijing, China). Vickers hardness test was performed with the micro Vickers indenter (height about 0.1 mm and diagonal 0.2 mm) on the specimens at 500 g load for 15 seconds, crosshead speed 0.015 mm/s. Penetration was performed in three different locations. To compare the study groups, the ANOVA (post hoc-Tukey) test was used for hardness testing analysis at a significant P-value of ($P \leq 0.05$).

3. RESULTS AND DISCUSSION

3.1. The SEM/EDX Studies

The SEM/EDX study element composition analyzes are revealed in Table 1. The particles of both VITA Lumex® AC and amber fine powder particles show irregular geometry with different particle sizes. The SEM images show the particle size of both VITA Lumex® AC and the amber fine resultant powder in this study, with an irregular geometry and different particle sizes.

Regardless of the percentages, the EDX test showed the same elements for both VITA Lumex® AC and the amber fine resultant powder, such as C, O, Na, Al, Si, K, Ca, and Cu, except for Mg, which was available only in the amber powder. This could highlight the use of amber glass powder safely in combination with VITA Lumex® as that of ceramic with zirconia of the same components [16]. The histogram of the amber fine powder and VITA Lumex® AC particle size distribution was examined by the Malvern Mastersizer analyzer.

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Table 1. Element composition of ceramic VITA Lumex® AC and study of Amber fine powder

No.	VITA Lumex® AC		Amber fine powder	
Elements	Weight %	Atomic %	Weight %	Atomic %
C	2.9	4.7	3.3	5.6
O	53.7	65.6	48.5	60.7
Na	6.4	5.4	7.9	6.9
Al	5.7	4.1	1.9	1.4
Si	24.5	17.0	29.0	20.7
K	4.3	2.2	0.6	0.3
Ca	1.0	0.5	5.5	2.7
Cu	1.5	0.5	2.0	0.6
Mg	0	0	1.1	1.3

The amber fine powder particle size mean value was 12.275 µm at a specific surface area of 0.391 m^2/g. The particle sizes fluctuated gradually between the d(0.1) of 2.440 µm and the d(0.9) of 37.002 µm. Yet, the mean particle size of VITA Lumex® AC was 26.672 µm at a specific surface area of 0.227 m^2/g. The particle sizes ranged between the d(0.1) of 4.985 µm and d (0.9) of 94.304µm, respectively, Fig. 2 and 3. The mean particle size histogram of amber fine powder was 12.275 µm, and the VITA Lumex® AC was 26.672 µm. This seems an acceptable particle size as the amber fine powder density involved within the VITA Lumex® AC powder density [12].

3.2. The DSC Studies

The DSC study materials test showed maximum enthalpy changes (ΔHd) during the endothermic event at a denaturation temperature (Td). The ΔHd (J/g)/Td (°C) were of 3095/130.18-196.42 for amber glass fine powder; 583.9/81.46-149.67 for VITA Lumex® AC; 765.6/125.78-191.45 for ceramic with 0.01 g of amber powder; 1346/118.75-185.63 for ceramic with 0.03 g of amber powder; and 2247/132.26-198.50 for ceramic with 0.05 g of amber powder, Fig 4. The amber resultant study fine powder showed extreme enthalpy changes (ΔHd) higher than VITA Lumex® AC ceramic.

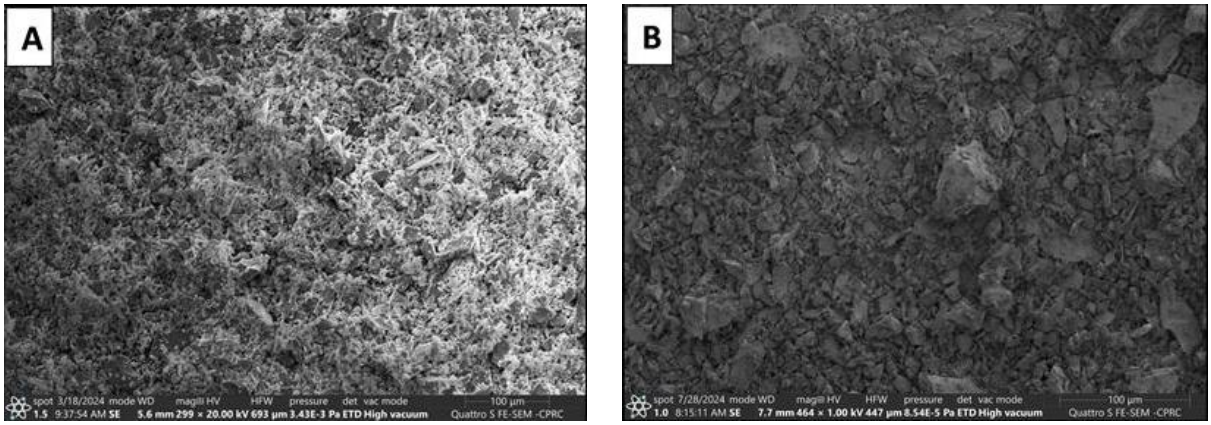

Fig. 2. SEM A, Study Amber resultant fine powder (12.275 µm); and B, VITA Lumex® AC powder (26.672 µm)

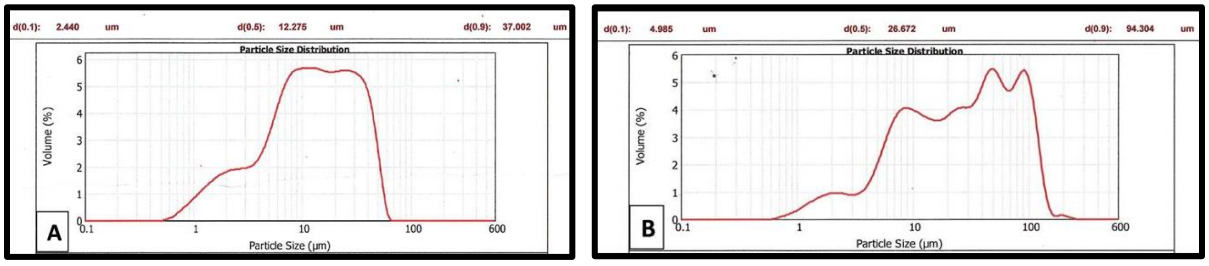

Fig. 3. Particle size distribution of A, Amber glass powder; B, VITA Lumex®

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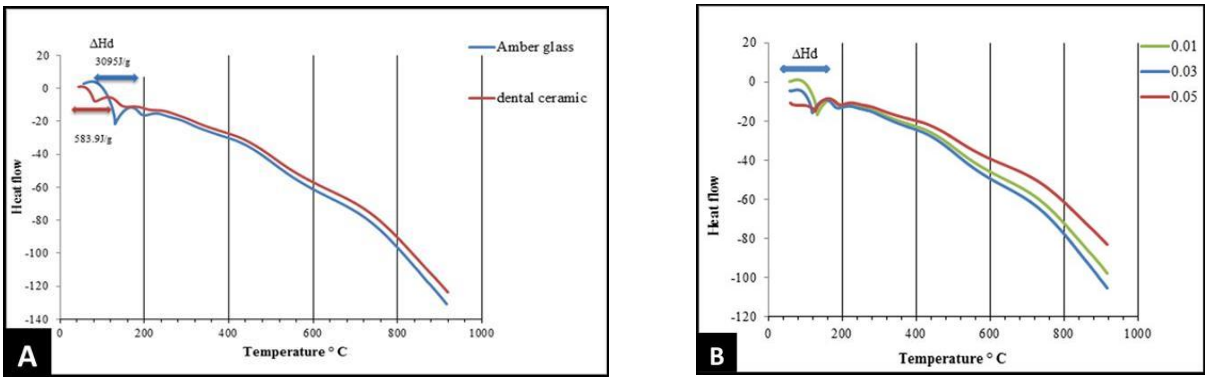

Fig. 4. Heat flow ΔHd determination using DSC A, amber powder vs. VITA Lumex® AC; and B, amber-ceramic study powders of different percentages of 0.01, 0.03, and 0.05 g

Yet, adding 0.01, 0.03, and 0.05 g of amber powder to Vita Ceramic gradually increased the VITA Lumex® ΔHd.This could be related to the high silica percentage of amber material in comparison to Vita ceramic, silica flow rate, which may agree with [27] who stated that more silica particle ratio within any composite material of different components could increase the slump flowing mass [27].

3.3. The Ion Release

The Cu and Al elements' ion release was measured by (AAS-GF). This technique is applied to quantify metal ions in very dilute concentrations in the range of parts per billion (ng/ml). Within the injection volume of 20 µl, the result of ions released from the media over 7 days after immersion of the specimens in artificial saliva was reported. The Cu/Al ion release values in ppb (ng/ml) were 1/3 for VITA Lumex® AC Ceramic; 2/3 for ceramic with an amber powder of 0.01 g; 1/1 for ceramic with an amber powder of 0.03 g; and 1/4 for ceramic with an amber powder of 0.05 g. Yet, the Cu and Al elements are essential in ceramics. However, their ion releases could affect human health [28]. These study materials, along with the three different additives, revealed that the Cu and Al ion release in ppb(ng/ml) is acceptable and below the hazard levels [30-32].

3.4. The XRD Analysis

Fig 5 demonstrates the amorphous structure of study materials using X-ray diffraction Analysis. The diffractogram for specimens with additives reflects a rise in amorphous phases as the amber powder ratio increased at an amorphous band of about 25-35°.Within an amorphous band of about 25-35°, the amber glass powder shows an amorphous structure similar to VITA Lumex® AC. Moreover, different amber additions' ceramic-amber material showed amorphous structures for both amber glass powder and Vita ceramics. This could be related to the compositional behaviour of the same elements as previously mentioned [5, 26]. Therefore, and according to the above findings, it could be concluded that amber glass could be considered acceptable for dental purposes. Consequently, the Vickers hardness is suggested to test the resultant ceramic-amber material for dental restorations.

3.5. The Vickers Hardness Studies

Table 2 shows that one-way ANOVA (post-hoc-Tukey) tests were performed to analyze the Vickers hardness mechanical property of the studied materials at a confidence level of 95% and significance of P≤ 0.05.

Table 2. Tukey's test shows the statistical analyzes of the studied groups

Groups		Mean Difference	Std. Error	P-Value	Sig.	95% Confidence Interval	
						Lower Bound	Lower Bound
G1	G2	-114.9033*	25.58079	.000	S	-183.7982	-46.0085
	G3	-146.3667*	25.58079	.000	S	-215.2615	-77.4718
	G4	-135.2233*	25.58079	.000	S	-204.1182	-66.3285
G2	G3	-31.4633	25.58079	.612	NS	-100.3582	37.4315
	G4	-20.3200	25.58079	.857	NS	-89.2149	48.5749
G3	G4	11.1433	25.58079	.972	NS	-57.7515	80.0382

G1: VITA Lumex® AC; C2: VITA Lumex® + 0.01 g amber; C3: VITA Lumex® + 0.03 g amber; C4: VITA Lumex® + 0.01 g amber

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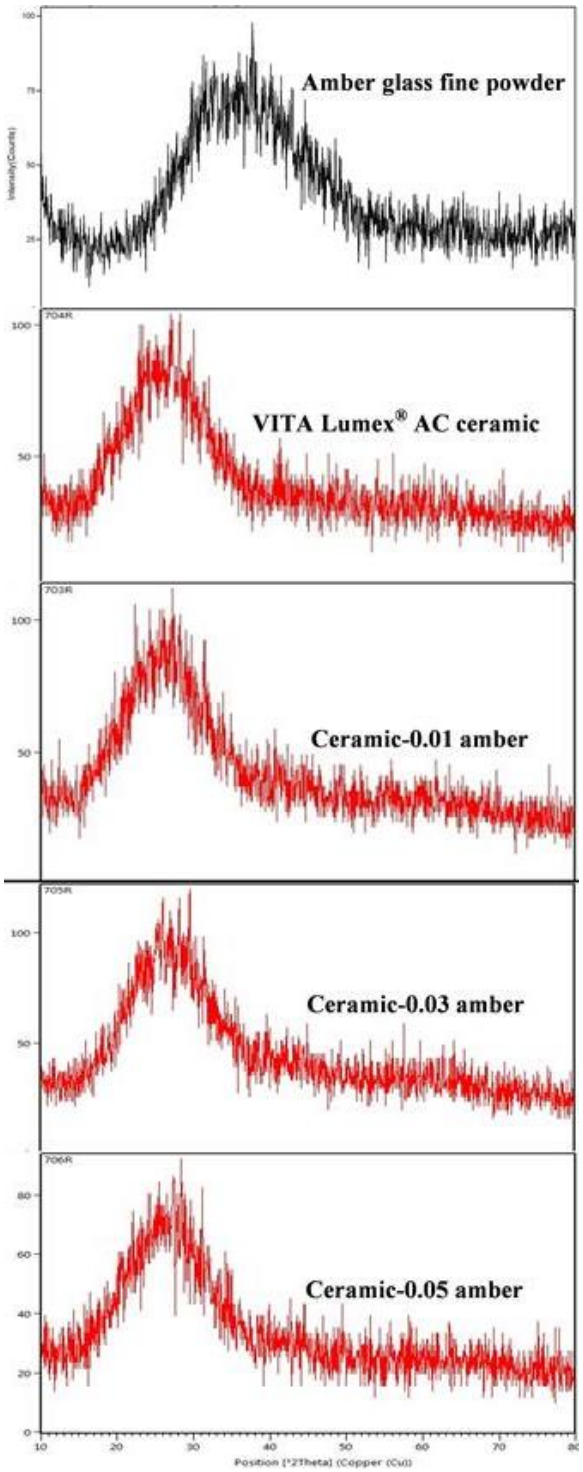

Fig. 5. The XRD analysis for studied materials

In terms of hardness property, the restorative material should withstand the indentation to avoid propagation up to fracture. In this study, the hardness of the experimental ceramic amber of three different additions of 0.01, 0.03, and 0.05 g showed a significant increase in HV than the VITA Lumex® AC. This could relate to the findings of this study the thermal behavior of amber glass powder alongside the high silica content in comparison to Vita ceramic. Also, this may be related to a smaller particle size of amber in this study powder, which in turn improves the density of resultant specimens [13].

4. CONCLUSIONS

Within the limitation of the present study, it concluded that the amber fine powder shows irregular geometry with different particle sizes such as ceramic powder, and it contains the same composed element as in ceramic such as C, O, Na, Al, Si, K, Ca, and Cu except that of Mg. in term of the particle sizes, the resultant amber glass powder of 12.275 µm was smaller than that of VITA ceramic of 26.672 µm. Moreover, a similar flow thermal behaviour was noticed regardless of the ΔHd (J/g) and Td (°C) of the tested amber, ceramic, and ceramic with amber additives of 0.01 g, 0.03 g, and 0.05 g. Yet, the Cu and Al element ions released in ceramic-amber material of different additions were reported to be acceptable in the range for human use. Furthermore, the amber, ceramic-amber material with different amber additions has shown an amorphous structure similar to Vita ceramics. Finally, the hardness of the ceramic amber of experimental specimens with three different amber additions of 0.01, 0.03, and 0.05 g shows higher HV hardness than the VITA ceramic.

To sum up, adhering to the above findings, the amber glass material could be considered as an additive agent for ceramics for dental purposes.

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