RESEARCH PAPER

In Vitro Evaluation of Ceramic-amber Hardness Supported Zirconia

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Abstrac*t: 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 dark amber glass behaviour as a new additive material for dental ceramics. The amber glass powder was prepared using the ball mill technique. For the amber glass powder characterization, 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 with an addition of 0.01g, 0.03g, and 0.05g 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 full 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 and 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

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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 which 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 ceramics properties or performance aspects. The ceramic hardness is typically affected by a function of both grain size with 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 oxides ceramics including MgO, BeO, Al2O3, MgAl2O4 and B4C 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 the temperature from 1100 to 1150°C. According to the general preparation for ceramics processing, the use of fine particles could result in higher densification and strength of sintered parts in comparison with 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 [13]. Nevertheless, research by Mei et al., shows that the Vickers hardness has a strong dependence 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].

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

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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 more micro silica particles to cement ratio from 2/1 to 2/2 the 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 which are 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) considered toxic [30, 31]. On the other hand, the total body burden of Al in

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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 study the characteristic behaviour of dark amber glass as an additive material for dental ceramics.

2. EXPERIMENTAL PROCEDURES

The pharmaceutical Amber glass (Omega 3, 200ml, 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 rotatory horizontal ceramic ball-mill machine for 18h (110rpm, CAPCO, UK). The ceramic balls were mostly circular with different sizes of 33-34, 26-28 and 22-25mm. The resultant powder was sieved using 45μm mesh grit at 10Hz for 2hr (Retsh W. EML, Germany). A high-energy vertical mini-lap planetary ball mill (Henan, Nanbei, NXQM-4L, China) was used for the resultant powder of fine particle sizes. 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 firstly 1:5 for 90min at 200 rpm, next continued with a ratio of 1:10 at 200 rpm for 7h. To avoid heat generation, an intermitted pausing of 10min was applied after each 30min of 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).

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

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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 were of this study fine amber glass powder. Ceramic VITA Lumex® AC powder, VITA Lumex® AC with three different amber glass powder additive ratios of 0.01g, 0.03g, and 0.05g using 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 silicone mould for study material of ceramic, ceramic-amber, and/or zirconia specimens with dimensions of 12×1.5mm in diameter and thickness respectively.

For the ion release test, the ceramic and/or ceramic-amber specimens of 1.5mm were prepared, and the Cu and Al released ions were measured after immersion 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%; KH $2PO_4$: 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).

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3.3. 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 0.1mm and diagonal 0.2mm) on the specimens at 500g load for 15 seconds, crosshead speed 0.015mm/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 ($P \leq 0.05$).

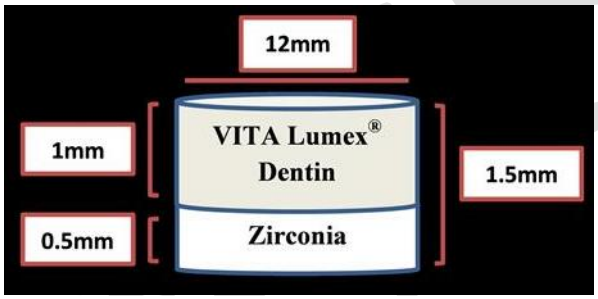

Fig. 1: Study specimens design

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 show irregular geometry with different particle sizes.

The SEM images show the particle size of both VITA Lumex® AC and this study amber fine resultant powder with an irregular geometry with different particle sizes.

Regardless of the percentages, the EDX test showed the same elements for both VITA Lumex® AC and this study amber fine resultant powder such as C, O, Na, Al, Si, K, Ca, and Cu, except the Mg which was available only in 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].

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

No.	VITA Lumex® AC		Amber fine powder	
Elements	**Weight %**	**Atomic %**	**Weight %**	**Atomic %**
C	2.9	4.7	3.3	5.6

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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 histogram of the amber fine powder and VITA Lumex® AC particle size distribution was examined by the Malvern Mastersizer analyzer. The amber fine powder particle size mean value was 12.275µm at a specific surface area of 0.391m^2/g. The particle sizes fluctuated gradually between the d(0.1) of 2.440µm and d(0.9) of 37.002µm. Yet, the mean particle size of VITA Lumex® AC was 26.672µm at a specific surface area of 0.227m^2/g. The particle sizes ranged between the d(0.1) of 4.985µm and d (0.9) of 94.304µm respectively, Fig 2 and 3.

The mean particle size histogram of amber fine powder was 12.275µm and the VITA Lumex® AC was 26.672µm. This seems an acceptable particle size as the amber fine powder density involved within the VITA Lumex® AC powder density [12].

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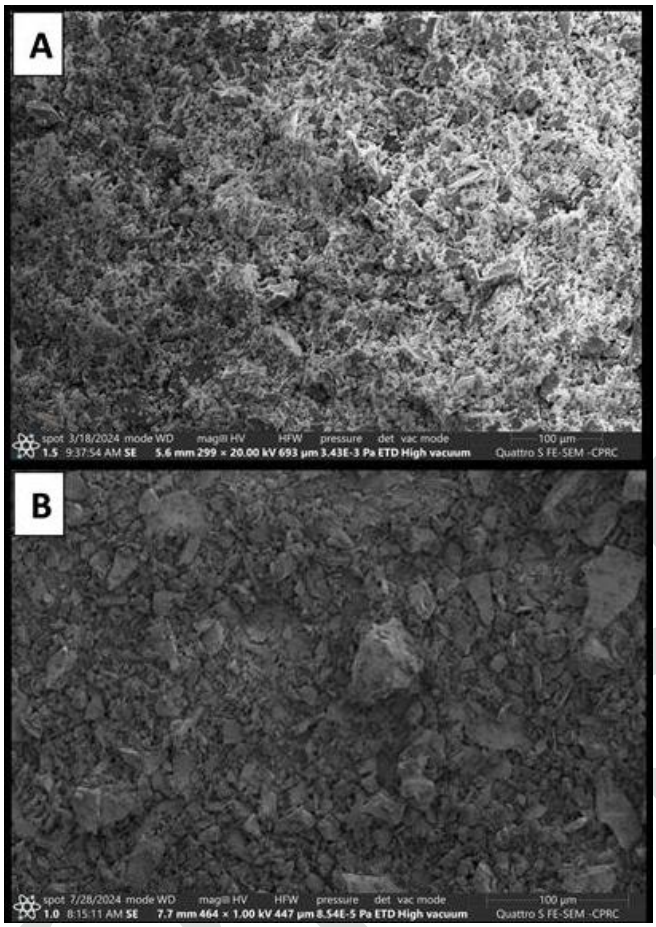

Fig. 2. SEM A, Study Amber resultant fine powder (12.275µm); and B, VITA Lumex® AC powder (26.672µm)

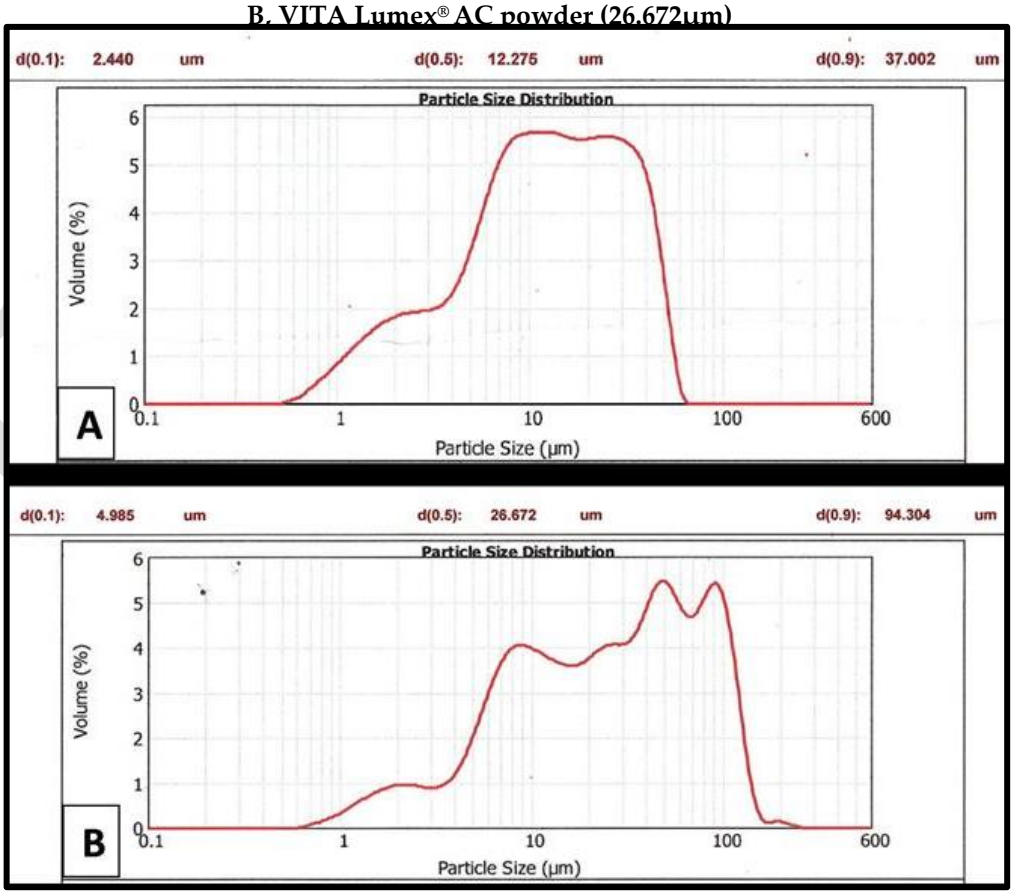

Fig. 3. Particle size distribution of A, Amber glass powder; B, VITA Lumex®

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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 0.01g of amber powder; 1346/ 118.75-185.63 for ceramic with 0.03g of amber powder; and 2247/132.26-198.50 for ceramic with 0.05g of amber powder, Fig 4. The amber resultant study fine powder showed extreme enthalpy changes (ΔHd) higher than VITA Lumex®AC ceramic. Yet, the addition of 0.01, 0.03, and 0.05g of amber powder to vita ceramic increased the VITA Lumex® ΔHd gradually. This could be related to the high silica percentage of amber material in comparison to Vita ceramic, silica flow rate, this 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].

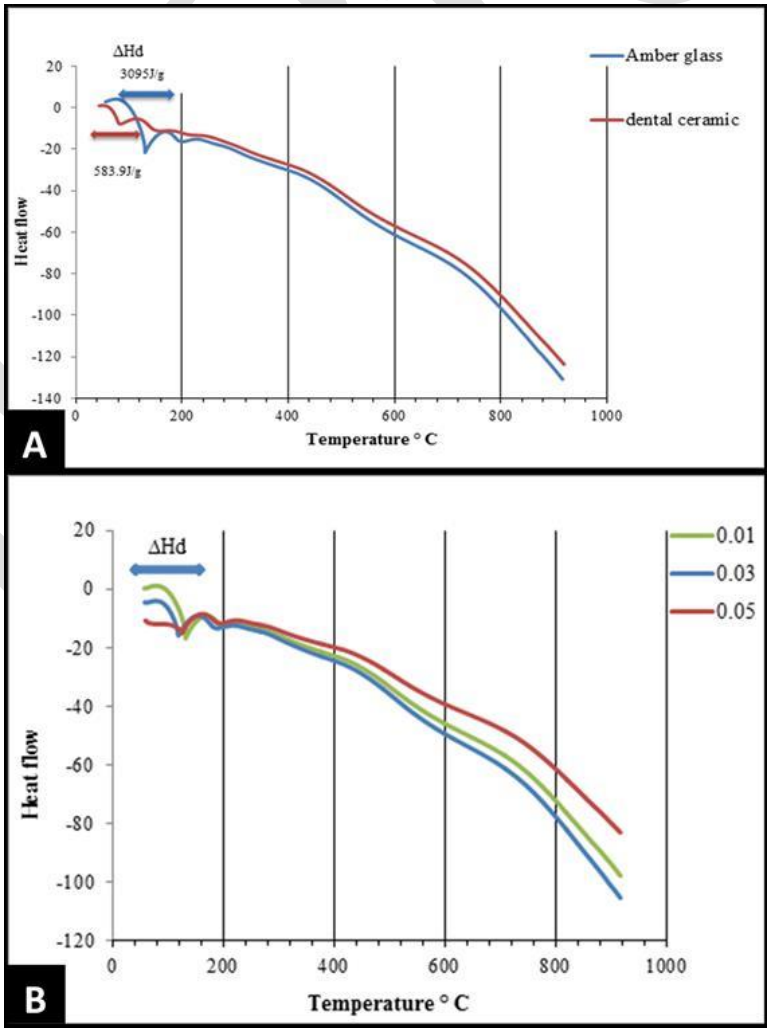

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Figure 4: Heat flow ΔHd determination using DSC A, amber powder vs. VITA Lumex® AC; and B, amber-ceramic study powders of different percentages of 0.01, 0.03, and 0.05g

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 20µl, 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 0.01g; 1/1 for ceramic with an amber powder of 0.03g; and 1/4 for ceramic with an amber powder of 0.05g. Yet, the Cu and Al elements are essential in ceramics. However, their ion releases could affect human health [28]. These study materials along 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 demonstrate the amorphous structure of study materials using X-ray diffraction Analysis. The diffractogram for specimens with additives demonstrates a rise in amorphous phases as an 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 that of VITA Lumex® AC. Moreover, the ceramic-amber material of different amber additions showed alike 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].

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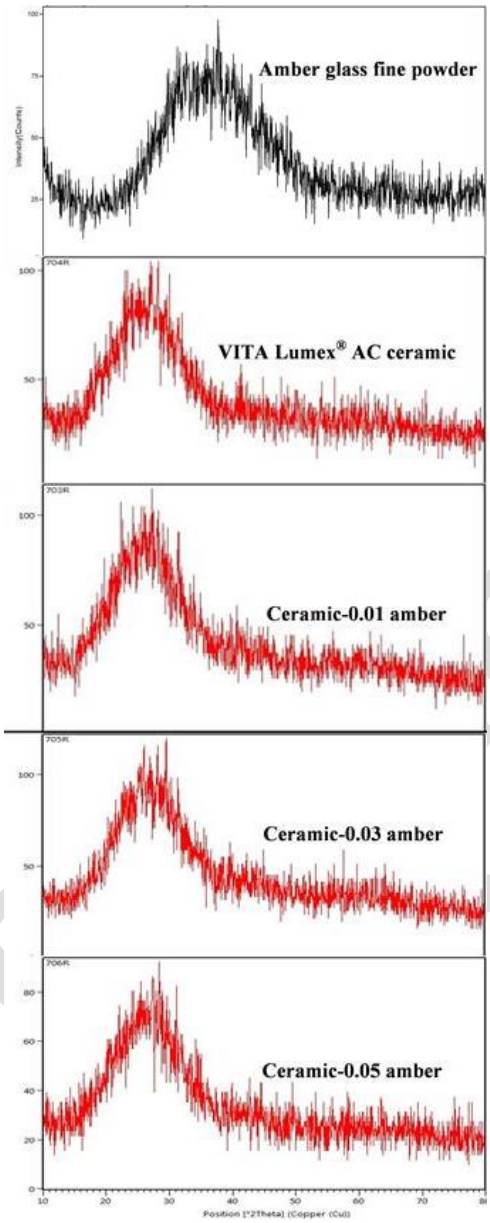

Figure 5: The XRD analysis for studied materials

Therefore, and according to the above findings, it could be concluded that the amber glass material could be considered an acceptable material for dental purposes. Consequently, the Vickers hardness is suggested to test the resultant ceramic-amber material for dental restorations.

3.4. The Vickers Hardness Studies

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

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

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 0.05g showed a significant increase in HV than the VITA Lumex® AC. This could relate to the findings of this study 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 12.275μm was smaller than that of VITA ceramic of 26.672μm. Moreover, regardless of the ΔHd (J/g) and Td (°C) of the tested amber, ceramic, and ceramic with amber additives of 0.01g, 0.03g, and 0.05g, a similar flow thermal behaviour was noticed. Yet, the Cu and Al elements ion released from this study ceramic-amber material of different additions were reported to be acceptable in range for human use. Furthermore, the amber, ceramic-amber material with different amber additions has shown an amorphous structure similar to vita ceramics.

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Finally, the hardness of the ceramic amber of experimented specimens with three different amber additions of 0.01, 0.03, and 0.05g show higher HV hardness than the VITA ceramic.

To sum up, and 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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