Mechanical Properties Improvement of Dental Amalgam Using TiO_2 and ZnO

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Abstract: In recent years, there have been many attempts to improve the properties of dental amalgam. The primary constituents are Ag, Sn, Cu, and sometimes Zn which were traditionally carefully chosen for certain specific characteristics. In this work, however, (0.5-1) % ZnO and TiO_2 were added separately and together. The addition was added to amalgam alloy powder and the prepared composite powder was triturated by a given percent of mercury. Wear test was performed with presence artificial saliva and conducted by Pin-on-Disc. Vickers hardness and compression strength was also conducted. The results showed that amalgam/ TiO_2 composite and amalgam/ hybrid composite posses the best wear resistance (85% compared to the control specimen), hardness (48% and 47% respectively with respect to the control specimen) and compression strength (63% and 62.5% respectively with respect to the control specimen).

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Key words*:* dental amalgam, TiO_2, ZnO, hybrid, wear resistance, Vickers hardness and compression strength.

1. Introduction

Amalgam is a metallic alloy formed by reaction between mercury and an alloy in powder form containing silver, tin, copper, zinc, etc. with a very complex metallurgical structure. Powder alloys can be classified into two groups: low-Cu content or conventional composition (5% or less copper) and high-Cu content (6 to 30% copper). Two different types of high-Cu alloy powders are available. The first is an admixed alloy powder and the second is a single-composition [1].

Dental amalgams have been used for dental restoration for more than 150 years because they are malleable, durable, and more affordable than gold or composites [2].

Up to now, there have been many attempts to improve properties of dental amalgam that these comprise treatment on amalgam alloy powder or addition some materials such as In, Au, Pa and organic materials. One of the latest additive materials is Palladium [3]. Another new additive materials was used Ag-Cu Nano-powder were investigated by Chung et al [4].

Yusif G. L. [5], used various fluids, such as natural saliva and fluoride to identified their effect on corrosion behaviour of high-Cu and low-Cu dental amalgam. He concluded that the high-Cu has more corrosion resistance and fluoride made the corrosion faster.

Tolou N. B. et al. [3], studied the fabrication and characterization of dental amalgams containing TiO_2 nanoparticles and evaluation of their compression strength, antibacterial and corrosion behaviour. The result suggested that amalgam/ TiO_2 NPs nanocomposite with 1 wt. % of TiO_2 NPs could be used as a biocompatible and bioactive dental material.

Yahya N.et al. [6], enhanced the hardness and other mechanical properties of dental amalgam material using zinc oxide and aluminium oxide nanoparticles as fillers. they used compressibility and Vickers micro hardness. The results show that there is an increment of 183% in hardness was observed by using zinc oxide nanoparticles and 229% by using aluminium oxide nanoparticles.

Jaber H. H. [7], explained the effect of adding Ti on corrosion resistance of high-Cu dental amalgam. Various tests, such as Tafel technique, Vickers hardness and microstructure observation have been used to measure the corrosion resistance, hardness and phases in the microstructure. it is found an improvement in corrosion resistance due to the form passive layer that increases with increasing titanium content. Hardness was also increased but there is no effect on the phases.

The American Dental Association (ADA) Council on Scientific Affairs had prepared charts comparing the important features of many of the popular direct and indirect restorative materials. The service life of dental restoratives depends on a number of factors one of them is a material. Material’s related factors include strength, hardness, toughness, and wear resistance [8].

The aim of this work is to investigate the effect of TiO_2 and/or ZnO composites on wear behaviour, Vickers hardness and compression strength of dental amalgam.

2. Experimental Procedure

2.1 Materials and specimen preparation

In the present study, the initial materials for making the dental amalgam included an admixed amalgam alloy consisting of high-cu (Rubygam Powder Amalgam) and amalgam alloy included low-Cu (Rubygam High Ng Powder Amalgam). It consists of micro-fine lathe-cut very thin needle-shaped cut particles.

Hg type SEPTODONT MERCURE was used in the amalgam, which is a confirmed material in dentistry with the highest purity (99.99%). The mercury to alloy powder ratio was (1.03:1) according to the manufacturer.

The size of TiO_2 particles was measured by Particle size analyzer was <80 µm, and the size of the ZnO was (80nm-20µm).The amounts of (0.5-1)% TiO_2 and/ or ZnO were added to amalgam alloy powder and weighting was done by a digital balance with 0.0001g accuracy. Also, two specimens were fabricated without additive high-Cu and low-Cu (as control amalgam). Then, this mixed powder was triturated by mercury in the time and frequency given by producer instruction via a digital capsule amalgamator (YDM-Pro).

The details of used materials are summarized in Table1.The amalgam paste was condensed into a steel die with a cylindrical container to form a specimen with the diameter and height of 4.0 ± 0.1mm and 8.0 ± 0.1mm, respectively. Specimens were hold in an incubator at 37 ± 1°C. The solution was used in the tests was synthetic saliva which closely resembles natural saliva, whose composition is shown in Table 2 [9]. The pH solution was 6.7 at 37Cº temperature.

2.2 Mechanical test

In order to measure the wear resistance, the required specimens were prepared and the specimens were stored in an incubator for 7 days at the temperature of 37 ± 1°C. The measurement of the wear was done using a micro-tester (Pin-on-Disk) MT 4003 version 10. It uses rotational method with a pin specimen loaded against a rotating disc. The experiments were done under wet conditions; saliva was used as fluid lubricant. The diameter of the circular wear track produced in the disk was 12.5 mm. In the present study, a stainless steel with R_a=0.265µm was used as a disc. The tests were performed using the load of 5 N, which was an appropriate choice considering a normal force on a real single tooth [10]. The disc was rotated at a constant speed of 150 rev/min [11].

The measurement of Vickers hardness was done by using a micro-hardness tester HVS 1000/China after 1 and 7 days from the end of preparation. The applied load was 500g for 15 seconds [12].

Measurement of compression strength was conducted by Instron machine type UNIVERSAL TESTING MACHINE/WDW 200, China with the speed of 0.5mm/min [13], at different times 1 hour, 24hours and 168hours (one week) from the end of trituration.

3. Results and discussion

Wear behaviour of the examined specimens was measured in terms of weight loss as shown in Figure (1). From the tribological point of view, the reason for a sharp rise in the weight loss within the initial cycles is expected by the process of asperity smoothing in the initial stage of contact. Lee [14] reported amalgam is a brittle material with limited capacity for plastic flow at room temperature.

It is well established that the wear rate depends on the normal load and the hardness or yield strength of the material. So, the lower wear resistance occur in the control amalgam. When increase copper content, wear resistance will be improved to about (40%) corresponding to the control specimen.

The addition of ZnO however caused clear improving in wear resistance (51%) corresponding to the control specimen. while the significant enhancement was associated by the addition of TiO_2 and same behaviour by hybrid addition, this can be attributed to the large difference between the hardness of the ZnO (HV=1500) and that of TiO_2 (HV=7800) [15]. Therefore, the hybrid and TiO_2 amalgams possess the best improving (about 85%) corresponding to the control specimen.

As shown in the Fig. (2) The hardness increases with time that the reason is the reaction rate which is quite slow and takes several days until week to reach its completion. This is reflected in the rate of the development of mechanical properties. In this time, there is residual mercury which is the weakest in each amalgams with low and high percent of copper [16]. It should be stressed here that high-Cu amalgams may also initially contain the $\gamma_2(Sn_{7\text{-}8}Hg)$ phase. In these amalgams, the elimination of γ_2 may occur over a substantial time period as the reactions which are diffusion controlled [17].

The results reveal that the minimum value of amalgams is 146 HV (control amalgam). It has been observed that the improving of HMV of high-Cu about (37%) corresponding to the control specimen, and when added 1% ZnO, the improving will reach to (40%). It can be seen that the improving of HMV is (48%) and (47%) when added 1% TiO_2 and hybrid, respectively.

Compression strength test (Fig. 3) conducted during this work also it supported the wear results almost in the same order as the hardness test. From the Fig., it is clear that the compression strength of control amalgam after one hour from the end of trituration is relatively low; this is because of the presence of mercury which is weaken the strength. The amalgamation reaction does not proceed enough to form the $\gamma_1(Ag_2Hg_3)$ and γ_2 phases during this period.

After 24 hours, however a significant improve in compression strength is obtained which reaches to(272 %), because a large amount of mercury was expected to be depleted by forming γ_1 and γ_2 phases.

After 168 hours (one week), amalgam reached its final strength, where mercury is depleted and transformed completely to γ_1 and γ_2 phases [18].

On the other hand, the compression strength of high-Cu between (1 hour and 24 hours) has the same improving in the control specimen (272 %). This can be attributed to the particles of low-Cu lathe-cut alloy initially reacted to form γ_1 and γ_2 phases.

It seems that γ_2 phase is an intermediate product which appears for a short time during setting. Since it reacts with copper coming from (Ag-Cu) eutectic to form finally η (Cu_6Sn_5) phase, it appears that the additives did not change the sequence of reactions of the amalgam.

The analysis reveals that, the improving in compression strength for high-Cu will reach to (41%) with respect to the control specimen measured after (168-hour). It was reported that [12] the increase of copper content in the amalgam powder results in changing the lathe-cut to spherical due to the deformation. This change in shape caused an increase in compression strength and hardness.

Further improvement was obtained when added 1% ZnO to reach (52%) compared to the control specimen because the addition of zinc oxide nanofiller into amalgam has improved its compressibility and hardness. It can be seen that the maximum improving of compression strength is (63%) and (62.5%) when added 1% TiO_2 and hybrid, respectively which due to the high hardness of TiO_2.

Table (1) details of used materials

Specimen No.	Ag wt.%	Cu wt.%	Sn wt.%	Zn wt.%	ZnO wt.%	TiO_2 wt.%
1	71	3.3	25.7	-	-	-
2	45	24	30.5	0.5	-	-
3	44.55	23.76	30.195	0.495	1	-
4	44.55	23.76	30.195	0.495	-	1
5	44.55	23.76	30.195	0.495	0.5	0.5

Table (2) Chemical composition of synthetic saliva [9]

No.	Constituent	gm/l
1	KCl	1.5
2	$NaHCO_3$	1.5
3	$NaH_2PO_4.H_2O$	0.5
4	KSCN	0.5
5	Lactic acid	0.9

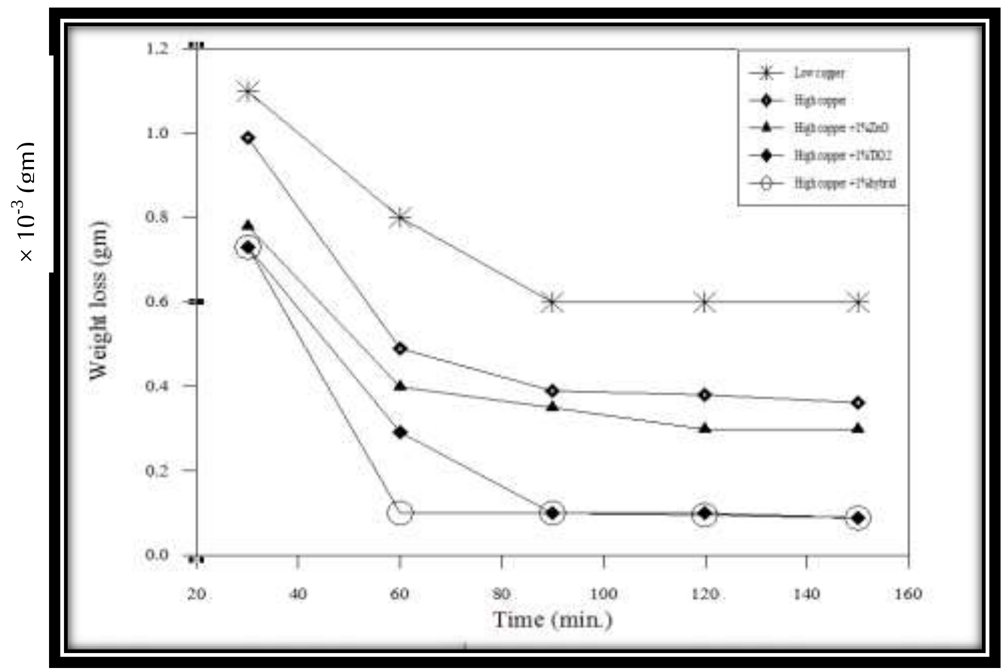

Fig.1 Weight loss as a function of exposure time for the various amalgams in saliva

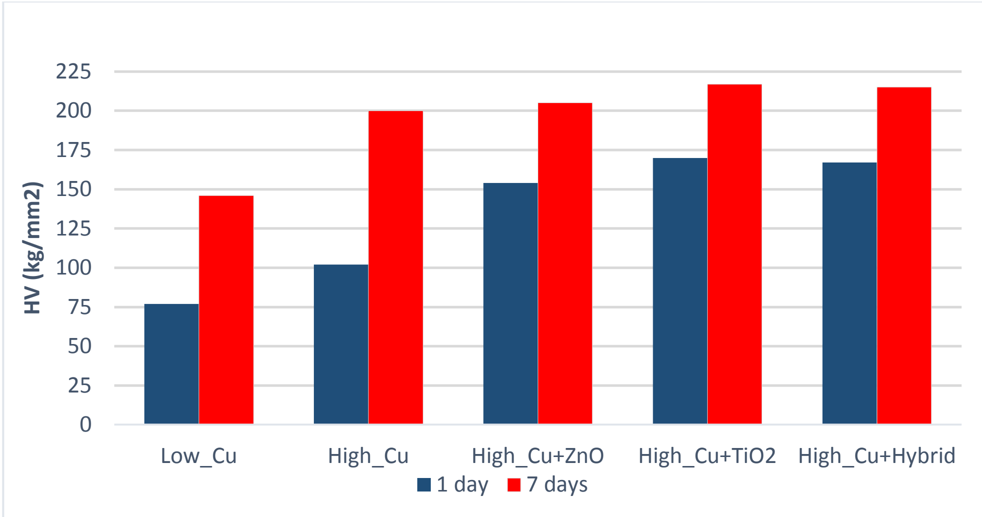

Fig.2 microhardness of dental amalgams

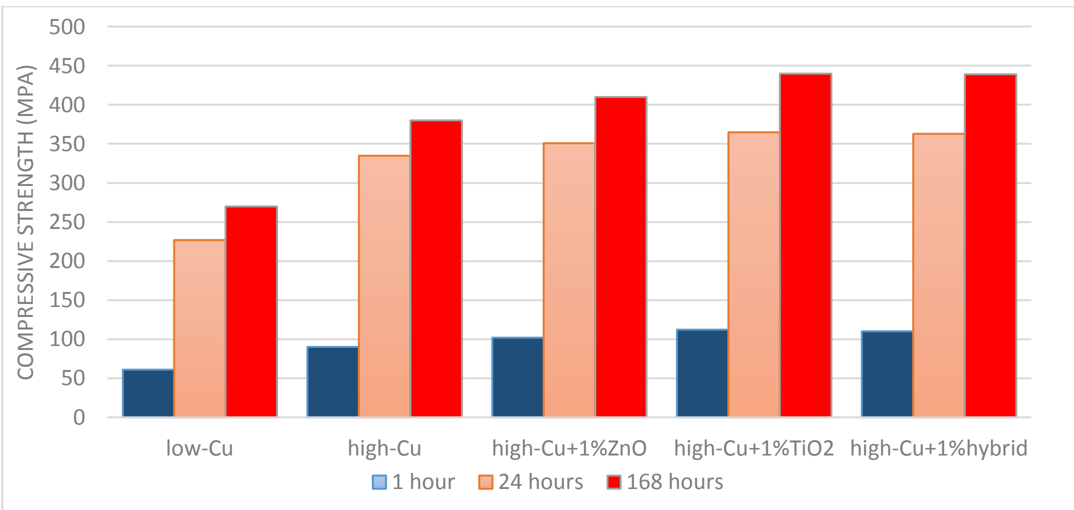

Fig.3 Compression strength at different aging time

4. Conclusion

1. Generally, wear resistance decrease with time.

2. Wear resistance was enhanced significantly in the hybrid and TiO_2 specimens, to reach (85%) compared to the control specimen.

3. An improvement in hardness was obtained in specimens with TiO_2 and hybrid (48% and 47%) respectively with respect to the control specimen.

4. The compression strength, however, was also improved in specimens with TiO_2 and hybrid (63% and 62.5%) respectively with respect to the control specimen.

5. The results of present study suggest that amalgam/ hybrid composite could be regarded as a dental material that provides better characters for dental applications.

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