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Enhancing the Mechanical and Physical Properties of Zirconium Dental Filling Material by Chemical Mixing with Polyvinyl Alcohol Polymer in Different Cure Times

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Abstract

Background: Zirconium is mainly composed of zirconia/silica particles. Zircon filling material is a light-curing, radiopaque nanohybrid composite resin designed for anterior and posterior tooth restorations with a high inorganic filler component. One synthetic polymer that dissolves in water is polyvinyl alcohol. Polyvinyl Alcohol's low toxicity, low propensity for protein adhesion, and biocompatibility make it useful in a range of medical applications. Contact lenses, eye drops, and cartilage substitutes are among the specific applications.

Objectives: evaluate the effect of chemical mixing polyvinyl alcohol polymer on the hardness, strength, water solubility, and water sorption properties of zircon dental filling material.

Patients and Methods: The biocomposite was prepared by dissolving 5g of polyvinyl alcohol in distilled water and stirring the mixture continuously for 30 minutes at 80°C. The mixture was stirred until a gel formed. Then, the mixed Universal Restorative material with zircon fillings was exposed to visible violet curing light for hardening, with different curing times (1, 2, 5, 10, 15, and 20 seconds). Then, we measured the strength, hardness, sorption, and solubility of the new mixed filling.

Results: Showed an increase in the hardness and the strength of the zircon filling materials after chemical mixing with Polyvinyl Alcohol polymer, compared with traditional zircon material that cured without Polyvinyl Alcohol polymer, and a decrease in the solubility and sorption of the filling material after chemical mixing.

Conclusion: An addition of polyvinyl Alcohol polymer particles to impact light-cure zircon dental filling material improves the impact physical and mechanical properties of the new mixed filling material.

Keywords: Zirconium, Nanohybrid, Polyvinyl alcohol polymer, Strength, Hardness, Sorption, Solubility.

Introduction

Zirconium, obscure before the late 1940s, became a significant engineering material for nuclear energy. Dental zircon has many advantageous properties, including biocompatibility and the ability to mimic the optical properties of real teeth (1, 2). A polymer nanocomposite is a blend of materials in which the filler is a nanomaterial and the matrix polymer. Adding nanofillers to the polymer matrix typically modifies the characteristics of the polymer (3). A restoration with full contour would exhibit

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excellent occlusal detail and a final shape, and a high translucency would enable the material to blend naturally with surrounding teeth (4). Due to their exceptional strength, monolithic zirconia crowns offer sufficient fracture resistance for dental crown restorations. Stress-induced transition toughening in stabilized zirconia is responsible for this resistance (5). Since zirconium does not absorb neutrons, it might be used as a perfect material in nuclear power plants (6). Zirconium metal is protected by a thin oxide layer, making it exceptionally resistant to corrosion by acids, alkalis, and seawater. For this reason, it is extensively used by the chemical industry. (7) ZrO2, or zirconia, is a wide bandgap metal oxide with potential applications in many scientific and technological domains. It exhibits admirable mechanical, optical, electrical, and thermal properties, including outstanding corrosion resistance, high fracture toughness, high hardness, a high refractive index, optical transparency, low thermal conductivity, and polymorphism features (8). One of the many polymer options is polyvinyl alcohol (PVA), a water-soluble polyhydroxy polymer characterized by its chemical structure, which features CH, CH2, and OH side groups. Due to its exceptional mechanical strength, biocompatibility, and non-toxicity, PVA has been widely investigated as an implant material in various biomedical applications, including drug delivery systems, dialysis membranes, wound dressings, artificial skin, cardiovascular devices, orthopedic implants, and maxillofacial surgeries when combined with ceramic materials (9, 10). Since poly PVA has great mechanical qualities, it is one of the more often used polymers. It is also biodegradable under the right circumstances (11). Handheld light-emitting devices, known as dental light curing units (LCUs), are used to cure photo-activated polymer-based restorative materials (PBRMs) (12). Using visible light energy, a photo initiator system is triggered in photo polymerization. This light-activated reaction produces reactive species (free radicals) that start the polymerization process by absorbing light photons (13, 14). To create a high-impact PVA/Zirconium nanocomposite, zirconium nanoparticles treated with polymers, specifically PVA, and cured for hardening filling material, were used in this study. The effects of this addition on various mechanical and physical properties over different curing times were also investigated. The objective of this study was to evaluate the impact of chemical mixing of PVA polymer on the hardness properties of zircon dental fillings.

Patients and Methods

Study design: The experiment was conducted at the University of Diyala, College of Science, from October 1, 2023, to March 1, 2024. The biocomposite was prepared by dissolving 5 g of PVA in distilled water, with continuous stirring for 30 minutes at 80°C. The mixture was then stirred continuously until gel formation occurred. We mix it with scope zircon fillings (3 M ESPER Filtek Z350 XT), approximately 5 g of universal restorative material. After mixing, the resulting materials were hardened using an O-Light device (Woodpecker O-Light unit, a product from DTE Woodpecker) with varying curing times (1, 2, 5, 10, 15, and 20 seconds) and a wavelength of light ranging from 420 nm to 490 nm. Additionally, the zirconium is hardened in two groups: a study group consisting of zirconium filling material mixed with PVA polymer, and a control group consisting of hardened zirconium filling material only. Then, the Shore-D device (Shore Durometer) was used to measure the hardness of the mixed and hardened materials. Fourier Transform Infrared Spectroscopy test (FTIR) was done to zirconium filling after mixing with the PVA polymer to evaluate the active groups. The PVA polymer imparted a strong rubber-like plasticity to the zirconium, and it hardened when exposed to an

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O-light device with a wavelength of light ranging from 420 nm to 490 nm, at varying time intervals. The test method includes placing the device perpendicular to the desired sample. Measure its hardness so that it is in contact with the surface of the sample whose hardness is to be measured, to insert the needle into the surface of the material, and for a waiting period of about three seconds, after which the hardness value is taken from the device. The applied pressure is in accordance with the specifications (DIN 53505) and equals 50 N, which is equivalent to 5 kPa. For Shore, several readings were taken at different places on the surface of the sample.

Strength was measured using a universal Instron testing machine. Each specimen was positioned on the bending fixture, and the load was applied with a crosshead speed of 1 mm/min by a rod placed centrally between the supports. Deflection was allowed to occur until fracture, and the scale of strength was recorded (15). Three square-shaped specimens, measuring 1.5 mm in thickness and 2 cm in size, were used to assess the solubility and water sorption of pure zirconium and a mixture of PVA and zirconium-based filling material. The specimens had one polished surface that was softening. For thirty minutes, the specimens were stored at room temperature. This weight figure was regarded as the specimen's starting weight (M1) Every day until a consistent weight (M2) was reached, all specimens were weighed using an analytical scale "Model JK-180; Chyo, Tokyo, Japan with an accuracy of 0.0001 g, in a water bath maintained at 37°C". The samples were weighed once again after being dried at 37°C in a vacuum oven to maintain their weight (M3). For every specimen, the values of water sorption (Wsp) and solubility (Wsl), expressed in µg/mm3, were computed using: $M2 - M3/V$ and $Wsl = M1 - M3/V$. The symbol V represents the volume of the specimen, in mm^3 (16). We followed the same steps as for the zirconium filling material, without mixing it with PVA, and measured it using all the tests applied to the zirconium with PVA. The results were recorded as a control group.

Statistical analysis

The data analysis was conducted using IBM SPSS 29 (IBM Statistical Package for the Social Sciences, version 29, Chicago, IL, USA). Descriptive statistics, including simple frequency and percentage calculations, were employed to present the data. Results were deemed non-statistically significant if the p-value exceeded 0.05. Conversely, p-values below this threshold were considered statistically significant, with those less than 0.01 classified as highly significant.

Results

Fourier transform infrared spectroscopy test (FTIR): The structures of the PVA polymer and zirconium were confirmed by Fourier Transform Infrared Spectroscopy test (FTIR). The FTIR spectrum showed absorption bands at 3309 cm^{-1} (OH) stretching (broad) of shellac, 2910.68 cm^{-1} (CH2) of aliphatic, 1730 cm^{-1} (C=O) of Ester, 1095 cm^{-1} (CO) Extended Ester, 1658 cm^{-1}(C=C) Shellac 1018 cm^{-1} (CO) (Extended Shellac) (Figure 1).

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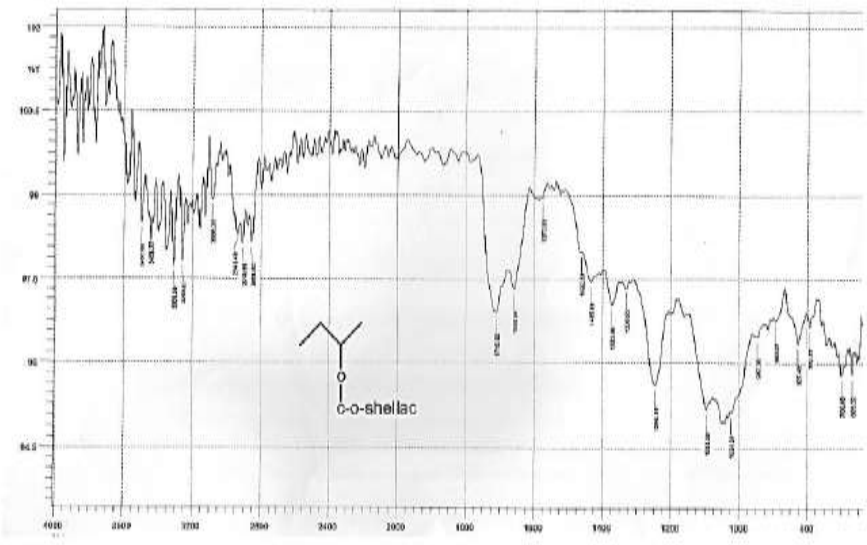

Figure 1. The FTIR test for zirconium filling material and PVA polymer.

The hardness: The results of the hardness show a high value for the study group compared to the control group. The groups' difference in hardness shows a highly significant difference in the study group at 10-15-20 sec curing time compared to the control group, which showed a non-significant difference at the same curing time. While the study showed a significant difference in the study group at 1-2-5 sec hardening or curing time compared to the control group, which showed a non-significant difference (Table 1).

Table 1. Descriptive statistics and groups' difference for the Hardness.

group	Time in sec	Descriptive statistics						Groups' difference		
		N	Mean	S.D	S.E	Min	Max	Mean difference	t-test	p-value
Control group	1	5	5.4	.60	1.34	4.000	7.000	6.013	-6.987	.076
	2	5	5.4	.67	1.51	3.000	7.000	6.544	-5.678	0.78
	5	5	6.8	.77	2.03	4.000	8.000	6.788	4.645	0.9
	10	5	7.4	1.12	2.50	5.000	10.000	8.800	-8.586	0.56
	15	5	8.8	2.02	2.70	6.000	11.000	8.999	-6.987	0.65
	20	5	9	3	3.3	8.000	13.000	9.987	-9.867	0.66
Study group	1	5	16.0	1.14	2.54	14.000	20.000	6.016	-11.345	.015*
	2	5	17.0	2.00	4.47	12.000	23.000	7.669	-12.45	.032*
	5	5	17.6	1.66	3.71	15.000	24.000	5.678	-13.456	.041*
	10	5	17.0	2.09	4.69	11.000	21.000	9.755	-12.456	.003**
	15	5	18.2	1.49	3.34	13.000	21.000	11.044	-13.542	.002**
	20	5	23.4	3.41	7.63	14.000	34.000	13.466	-14.453	.001**

The strength: The results of strength represented a high value in the study group that mixed zirconium with PVA, compared to the control group that used pure zirconium (Table 2). It showed a highly significant difference in the study group at 1-5-10-20 sec curing time, and showed a significant difference in the study group at 2-5 sec curing time, and showed a non-significant difference in the control group at (1-2-5-10-15-20) sec curing time.

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Table 2. Descriptive statistics and groups' differences for the strength (N/mm2).

group	Time in sec	Descriptive statistics						Groups' difference		
		N	Mean	S.D	S.E	Min	Max	Mean difference	t-test	p-value
Control group	1	5	99.654	4.64	0.34	79.000	104.000	8.099	-6.654	.096
	2	5	97.875	4.66	1.58	63.000	102.000	7.146	-4.118	0.38
	5	5	93.875	2.87	2.93	94.000	101.000	6.987	-6.633	0.66
	10	5	121.789	11. 2	1.50	85.000	140.000	8.895	-7.006	0.50
	15	5	88.974	12. 2	2.79	66.000	151.000	8.326	-8.337	0.25
	20	5	101.432	3.87	2.3	84.000	133.000	9.976	-9.212	0.16
Study group	1	5	133.453	3.14	3.53	104.000	240.000	9.916	-11.345	.005**
	2	5	144.432	5.05	4.44	120.000	293.000	10.459	-12.45	.002*
	5	5	157.624	7.60	2.72	105.000	254.000	9.078	-13.456	.031*
	10	5	187.011	4.09	3.99	131.000	210.000	11.795	-12.456	.000**
	15	5	188.232	3.99	4.39	163.000	210.000	13.004	-13.542	.002**
	20	5	203.411	7.41	6.63	188.000	304.000	13.065	-14.453	.001**

Water sorption: The results of the Water sorption of the zirconium mixed with PVA decreased in the study group compared to the control group, as shown in Table 3. It was demonstrated that water sorption results show a significant difference in the study group at all curing times (1-2-5-10-15-20 sec), while showing a non-significant difference in the control group at all curing time intervals.

Table 3. Descriptive statistics and groups' difference for the Water sorption (mg/cm2).

group	Time in sec	Descriptive statistics						Groups' difference		
		N	Mean	S.D	S.E	Min	Max	Mean difference	t-test	p-value
Control group	1	5	0.468	0.019	0.006	0.433	0.502	0.280	32.155	.196
	2	5	0.485	0.021	0.004	0.455	0.500	0.342	33.456	0.33
	5	5	0.534	0.022	0.003	0.590	0.585	0.345	23.456	0.66
	10	5	0.543	0.31	0.004	0.499	0.560	0.432	32.433	0.54
	15	5	0.541	0.30	0.004	0.489	0.555	0.234	30.554	0.55
	20	5	0.431	0.231	0.005	0.389	0.470	0.295	23.456	0.56
Study group	1	5	0.355	0.366	0.006	0.302	0.387	0.294	34.567	.035*
	2	5	0.432	0.905	0.006	0.402	0.460	10.459	12.45	.052*
	5	5	0.324	0.460	0.005	0.312	0.356	9.078	13.456	.038*
	10	5	0.311	0.309	0.007	0.288	0.345	11.795	12.456	.050*
	15	5	0.232	0.299	0.005	0.202	0.260	13.004	13.542	.042*
	20	5	0.311	0.341	0.006	0.279	0.389	13.065	14.453	.021*

Water solubility: The results show that the Water solubility of zirconium mixed with PVA decreases in the study group compared to the control group at the same curing time, as shown in Table 4. The results represent water sorption data that show a highly significant difference in the study group at all curing times (1-2-5-10-15-20 sec), while showing a non-significant difference in the control group at all time intervals.

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Table 4. Descriptive statistics and groups' difference for the Water solubility (mg/cm2).

group	Time in sec	Descriptive statistics						Groups' difference		
		N	Mean	S.D	S.E	Min	Max	Mean difference	t-test	p-value
Control group	1	5	0.079	0.008	0.006	0.043	0.072	0.068	7.055	.196
	2	5	0.060	0.008	0.006	0.045	0.070	0.054	7.956	0.33
	5	5	0.086	0.006	0.005	0.050	0.0 85	0.064	8.456	0.66
	10	5	0.071	0.007	0.006	0.049	0.060	0.061	9.003	0.54
	15	5	0.096	0.006	0.006	0.069	0.105	0.075	6.054	0.55
	20	5	0.071	0.007	0.005	0.039	0.060	0.064	7.956	0.56
Study group	1	5	0.021	0.003	0.005	0.012	0.037	0.021	4.067	.000**
	2	5	0.030	0.004	0.004	0.022	0.040	0.023	4.045	.000**
	5	5	0.031	0.002	0.005	0.022	0.050	0.021	5.006	.000**
	10	5	0.029	0.004	0.003	0.018	0.035	0.025	3.406	.000**
	15	5	0.029	0.003	0.004	0.022	0.032	0.021	3.042	.000**
	20	5	0.030	0.002	0.004	0.021	0.039	0.022	3.153	.000**

Discussion

The present study showed an increase in the hardness and strength of the zirconium filling material after mixing with PVA in most curing times, compared to pure zirconium. Conversely, it also showed a decrease in water sorption and solubility of the zirconium filling material after mixing with PVA in most curing times, compared to pure zirconium. The results of the present study indicate a significant increase in the hardness of materials due to the addition of PVA to ZrO2 nanoparticles, which have a more uniform surface and a higher accumulation of nanoparticle material, as noted by Hudson et al. (15), resulting in a significantly larger filler content. The current study's findings indicate that adding zirconium oxide to high-impact PVA significantly increases impact hardness, particularly with curing times of 1-2–5 seconds, and exhibit highly significant differences at 10-15–20 seconds. When Al-Hiloh et al. (16) used zerconia nanoparticles with traditional heat-cured denture base resin, he obtained similar results. Safi (18) also agree with the hardness result when using heat-cured acrylic denture bases and the addition of silanized nano-ZrO2 fillers, which showed an increase in the hardness of the zirconium filling material. The results of the strength tests of the zirconium show high significant difference at 1-10-15-20 sec than the 2-5 sec that show significant difference, Due to the formation of supra-molecular bonds or cross-links that envelop or shield the nano-fillers and impede the spread of fractures, the interfacial shear strength between the zirconium filler and PVA is high, contributing to the increase in strength of the zirconium dental filling. Additionally, a strong link between the dental filling material (zirconium and PVA) can alter the development of cracks. This finding agrees with Al-Hiloh A.,and Sun L., et al. (16, 19).

The diffusion property of water molecules through the mixture of zirconium dental material and PVA is significantly lower at all interval curing time than that control group of pure zirconium dental material without PVA mixing, the reduction in water sorption may be caused by decrease surface roughness of

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zirconium dental filling material by PVA partials due to the PVA particles have very small size and well dispersion, and will be improve the surface of the specimen and also replacing the hydrophilic resin of the zirconium dental material particles. Mohammed (20) concurred with the study's findings, which claimed that a rise in the proportion of ZrO2 nanofillers reduced water sorption. The absorbed polymer's total volume may be decreased by the ZrO2 nanoparticles, which are insoluble in water, as Ferracane J (21) and Panyayong, W., et al. (22).

The result of water solubility showed a significant decrease in the study group compared to the control group. The reason for the decrease in water solubility might be attributed to the insoluble nature of the zirconium filler material. Consequently, the inclusion of PVA as an additive in the specimen mass would lower the overall solubility of the mixture. The findings of this investigation agreed with those of Gurbuz O and Pinarkursoglu F. (23), who discovered that the solubility of acrylic resin decreased upon the addition of milled glass fiber fillers. The findings also supported and agreed with Mohammed's (20) claim that water solubility reduced as the percentage of ZrO2 nanofillers increased. Furthermore, the current results agree with those of Al-Hiloh A. (16), which indicate that the addition of ZrO2 to the acrylic matrix improves and decreases the water solubility of ZrO2. Recently, the use of nanomaterials has increased in the fields of physics and microbiology due to the strength and effectiveness of these materials and the spread of their uses in most fields, as seen in (24), which uses TiO2 in microbiological applications. In addition, the uses of nanomaterials were not limited to increasing physical properties, such as hardness; chemicals were also used to enhance the hardness of living tissues, including bones, by using bisphosphonates to heal and increase bone strength (25).

Conclusions

Numerous physical features of ZrO2 Nanoparticles, which are represented by zirconium dental materials, are improved by the addition of polyvinyl alcohol, or PVA. When zirconium and PVA are mixed, the new mixing filling material's strength and impact hardness of modified zirconium rise. At the same time, the solubility and water sorption of the mixing filling material decrease. PVA material is used to enhance the properties of the zirconium dental material. Therefore, it can be used with other filling materials to produce new mixtures with new physical and chemical properties, or with the same filling material at different concentrations to achieve other desirable results.

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Ethical clearance: This study was approved by the Research Ethical Committee of College of Medicine/ University of Diyala (No:2024MAH873).

Conflict of interest: None.

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تحسين الخواص الميكانيكية والفيزيائية لمادة حشو الأسنان الزركونيوم عن طريق الخلط الكيميائي مع بوليمر كحول البولي فينيل في أوقات معالجة مختلفة

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الملخص

الخلفية: يتكون الزركونيوم بشكل رئيسي من جزيئات الزركونيا/السيليكا. مادة حشو الزركون هي راتنج مركب نانوي هجين، مقاوم للأشعة فوق البنفسجية، مقاوم للتصلب بالضوء، مصمم لترميم الأسنان الأمامية والخلفية، ويحتوي على نسبة عالية من الحشو غير العضوي. أحد البوليمرات الاصطناعية التي تذوب في الماء هو كحول البولي فينيل. سمية كحول البولي فينيل المنخفضة، وقابليته المنخفضة للالتصاق بالبروتين، وتوافقه الحيوي، تجعله مفيدًا في مجموعة من التطبيقات الطبية. من بين التطبيقات المحددة العدسات اللاصقة، وقطرات العين، وبدائل الغضاريف.

الأهداف: تقييم تأثير الخلط الكيميائي لبوليمر كحول البولي فينيل على صلابة مادة حشو الأسنان الزركونية، ومتانتها، وقابليتها للذوبان في الماء، وخصائص امتصاص الماء.

المرضى والطرق: تم تحضير المركب الحيوي عن طريق إذابة ٥ غرامات من كحول البولي فينيل في الماء المقطر وتحريك الخليط باستمرار لمدة ٣٠ دقيقة عند درجة حرارة ٨٠ درجة مئوية. تم تحريك الخليط حتى تشكل هلام. بعد ذلك، عُرضت مادة الترميم العالمية المختلطة مع حشوات الزركون لضوء معالجة بنفسجي مرئي للتصلب، بأزمنة معالجة مختلفة (١، ٢، ٥، ١٠، ١٥، و٢٠ ثانية). بعد ذلك، قيست قوة وصلابة وامتصاص وذوبان الحشوة المختلطة الجديدة.

النتائج: أظهرت زيادة في صلابة ومتانة مواد حشو الزركون بعد الخلط الكيميائي مع بوليمر كحول البولي فينيل، مقارنةً بمادة الزركون التقليدية التي تصلب بدون بوليمر كحول البولي فينيل، وانخفاضًا في ذوبان وامتصاص مادة الحشوة بعد الخلط الكيميائي.

الاستنتاج: إضافة جزيئات بوليمر كحول البولي فينيل إلى مادة حشو الأسنان الزركونية المعالجة بالضوء تُحسّن الخصائص الفيزيائية والميكانيكية للحشوة المختلطة الجديدة.

الكلمات المفتاحية: الزركونيوم، النانو هجين، بوليمر كحول البولي فينيل، القوة، الصلابة، الامتصاص، والذوبان.

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