Redesign of Zinc Polycarboxylate Dental Cement

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Abstract

The formulations and the properties of the Zinc Polycarboxylate Dental Cements are reviewed then new cements of this type are prepared with modified solid part of the cement. High silica glass powder with different particle sizes is prepared and added with different weight percentages to the zinc oxide. The liquid part of the prepared cements was merely 44% concentration of the polyacrylic acid. Accordingly, the usual and expensive additives to the liquid and solid part of the cement are eliminated. The working and setting times, compressive strength, flexural strength and modulus are measured according to ADA specifications. The formulated cement has long working times without much lengthening of the setting times. In addition, the mechanical properties have noticeably improved as compared with the original cements.

Keywords: Zinc Polycarboxylate Cements, Working and Setting Times, Compressive Strength, Flexural Strength and Modulus

Introduction

Materials design is a continuing and active field in dentistry. The development in this field includes metals and alloys, ceramics and glasses, natural and synthetic polymers, and composite materials.

Cements are essentially ceramic materials; in which the ceramic powders convert into solid via a chemical reaction. Inorganic or organic acids initiate setting reaction in the case of dental cements; which found a variety of applications in dentistry. The main utilizations of cements in dentistry are for cavity lining, luting applications and as more dedicated products for sealing root canals as part of a course of endodontic treatment [1].

In the early 20th century; zinc oxide-phosphoric acid, zinc oxide-eugenol and silicate glass-phosphoric acid cements were discovered. These zinc phosphate, zinc eugenate, and silicate cements were widely used until the 1970s, when new cements began to be developed [2]. Currently, many groups of cements are employed in dentistry; however, two prime groups of cements that are based on polyacids are widely utilized. The first relies on the reaction between zinc oxide and the molecules of a polyacid, which is called the polycarboxylate cements. The second group utilizes cations released from ion-leachable glass that reacts with the polyacid, called the glass ionomer or polyalkenoate cements. The first group have resin matrix and left unreacted zinc oxide particle, whereas, the glass ionomers has ceramic matrix composite systems. Recently, a third group is developed which may be viewed as hybrids of the first and the second group of cements [1].

Zinc polycarboxylate cements (ZPC) are preferred for luting fixed restorations because they reveal good pulp compatibility. These cements did not cause initial pain that takes place after luting when cast restorations were inserted with polycarboxylate cements; because they did not cause a severe reaction. However, these cements shrink more extensively than other materials such as zinc phosphate cements. This shrinkage, due to cross-linking of polymer chains with zinc atoms, possibly affects the integrity of bulk restorations. [3]

The ZPC contains zinc oxide powder similar to zinc phosphate cement except in that it utilizes a water-soluble polymeric acid in place of phosphoric acid. This makes the ZPC capable to adhere to dentine and enamel; thus, these cements are known as the first adhesive cements. The advent of ZPC was certainly, one of the significant developments in the history of the adhesive dental materials [4]. The prime motivation for the development of ZPC is the need to combine both the strength of zinc phosphate cements with the adhesiveness and biocompatibility of the so-called Zinc Oxide Eugenol cements (ZOE cements). In general, ZPC shows only low and short-term cytotoxicity, also, it shows the capability for establishing chemical interaction with hydroxyapatite of the dentine [5].

The zinc oxide reacts with the polyacrylic acid (also called polycarboxylic acid) forming a cross-linked structure of zinc polycarboxylate [6]. The set cement consists of the residual zinc oxide particles bonded together by this amorphous gel-like matrix. The cementing mix is more viscous than zinc phosphate mix, but because of its different rheology, it flows adequately under pressure. By comparison with the glass ionomer cements, the ZPC is more plastic and reaches full strength more rapidly [2].

Additionally, the dentist is much aware about the working and setting times of the cements. The working time is measured as the time from the start of mixing to the maximum time the cement can be manipulated. i.e. the viscosity of the cement mix is still low enough to flow readily under pressure to form a thin film. In other words, the setting time refers to the period during which the matrix formation has reached a point at which an external physical disturbance will not cause permanent dimensional changes. The setting time should be reasonably long so final finishing procedures associated with the restoration can be accomplished [7].

The rate of setting of ZPC is affected by the powder/liquid ratio and the reactivity of the zinc oxide. It is also affected by the particle size, the presence of additives, and the molecular weight and concentration of the polyacrylic acid. At luting consistency (i.e. uniformity and regularity), the recommended powder/liquid ratio is about 1.5:1 by weight. The working time is 2.5 to 3.5 minutes at room temperature, and the setting time is 6 to 9 minutes at 37°C. The ZPC flows under pressure out to the same degree of zinc phosphate mix and forms film thickness of 25 to 35 μm. Nevertheless, the short working times of the ZPC have been recognized as a potential problem. This has been overcomed by the recent formulations via adding the amounts of one or more of other unsaturated carboxylic acids like tartaric acid, itaconic acid and maleic acid to the polyacid liquid. These acids have a property of extending the working time without markedly affecting the setting time of the cement. The ability to extend the working time is particularly useful; especially for mixes that have a higher powder liquid ratio when they are being used as cavity bases [4, 8].

The powder of the ZPC is the zinc oxide, in some brands, contains reinforcing fillers such as 1% to 5% tin, magnesium oxide, or 10% to 40% aluminum oxide. The powder mix usually fired and ground to reduce the reactivity of the zinc oxide. A small percentage of stannous or other fluoride may also be included to improve mechanical properties and provide leachable fluoride. The liquid is approximately a 40% aqueous solution of polyacrylic with other organic acids that extend the working time. The molecular weight of the polymer is generally in the range of 30,000 to 50,000, which accounts for the viscous nature of the solution. In some brands of these cements, the polyacrylic acid component is dried and added to the powder to aid accurate weight ratio. Small amounts of NaH_2PO_4 may be added to the liquid to both reduces the viscosity of the polyacrylic acid and retards the setting of the cement. The manufacturer also controls the viscosity of the cement liquid by varying the concentration, the molecular weight of the polymer or by adjusting the pH by adding sodium hydroxide [2, 8, 9].

The compressive strength of these cements is in the range of 55-85 MPa, the tensile strength is 8-12 MPa, and Modulus of elasticity is 4-6 GPa. Strength increases with the powder/liquid ratio, reaching a maximum ratio at about 2:1 by weight, and it is increased also by additives such as alumina. In general, these cements have somewhat lower compressive strengths than zinc phosphate cements but are significantly stronger in tension. The lower compressive strength of the ZPC compared with the zinc phosphate cement is not regarded as a drawback for two reasons. The first is that the compressive strength of the ZPC is still in the acceptable region. The second reason is that higher compressive strength is usually at the expense of the diametral and flexural strength. The cement gains strengths rapidly after the initial setting period; the strength at 1 hour is about 80% of the 24-hour value. Long-term storage in water does not appear to have an adverse effect on the mechanical properties. The ZPC have superior properties on the other cements in that they have some distinctly plastic character in their behavior. This behavior can be modeled as that of a thermoplastic composite, which implies that there are only weak cross-links between the polymer molecules [2, 8, 9].

Another comparison between the zinc phosphate, polycarboxylate, and glass ionomer cements is done by Duymus and coworkers [10]. They found that the max temperature of the exothermic peak of the setting reaction of the zinc phosphate was increased when the liquid ratio was increased. Quite the opposite, that temperature is increased with the increase of powder ration for the ZPC and the glass ionomer cements.

The properties of the ZPC are continuing research and developments area. The effects of ultrasound on the setting reaction of ZPC have been studied by Shahid and coworkers [11]. Utilizing FTIR spectra, the ratios of absorbance peak height of the reacted cement at $1400 cm^{-1}$ $_-COO^-$ to that of the unreacted portion at $1630 cm^{-1}$ $_-COOH$ were measured for cements treated with ultrasound. These rations then compared with those obtained for the untreated cement with ultrasound. They have found that treating the cements with ultrasound for 15 s after 60 s from start of mixing enhances the setting reaction. This may be due to an increase in powder surface area by breaking up aggregates of the solid particles. The author concludes that the enhanced setting via ultrasound can allow ZPC to be used as a root end filling material and for bonding orthodontic brackets.

Karimi and coworkers [12] prepared ZnO/MgO nanopowders with spongy morphology by a sol-gel technique and constant frequency ultrasonic waves (sonochemical method). They show that this type of powders improves the compressive strengths of the prepared ZPC at an appropriate powder/liquid ratio.

The selection criteria for dental cements are thoroughly investigated by Pameijer [13]. The ZPC is continually highly ranked between other cements due to their high biocompatibility and the property of little or no irritation to the pulp, even at a remaining dentin thickness of 0.2mm, which is explained in terms of that the long molecular chains of the polyacrylic acid prevent penetration into the dentinal tubules.

Fraunhofer [14] shows that the optimal bonding between dental cements and substrate surface is due to combined effects of specific and mechanical adhesion. The specific adhesion is achieved through molecular interactions between the adhesive and the substrate surface. The cross-linking of short chain molecules led to the formation of 3-dimensional networks of molecular chains. Zinc phosphate cements show only mechanical adhesion through interlocking at the interface with the restoration and that with the tooth. On the other hand, ZPC exhibits both mechanical adhesion and some chemical bonding.

The seepage of oral fluids containing bacteria and debris between the tooth and restoration or cement layer is referred as microleakage. The microleakage arises from the lack of adhesion of the luting cements to the tooth structure. Mirkarimi and coworkers examined the microleakage of steel crowns for zinc phosphate cement, glass Ionomer cement, and ZPC [15]. The results show that 45% of specimens of zinc phosphate and 5% of that of the glass ionomer showed leakage extending on to occlusal aspect. In contrary, none of the specimens of ZPC shows that disadvantage.

Recently, the effects of addition of calcium hydroxide to ZPC powder have been studied by Zamanian and coworkers [16]. They found that the setting time of the resultant cements is increased with the increase in the calcium hydroxide content. In addition, the compressive strength of the cements has their optimum with the addition of 5wt% of calcium hydroxide.

Summarizing, the ZPC is expected to continue engaging as dental cement due to its favorable properties and R&D activities will continue to improve its properties. The working time and setting time of the ZPC are dependent on the same chemical reaction, but they are manipulated through the reactivity and the particle size of the zinc oxide, the presence of additives, molecular weight and concentration of the polyacrylic acid and the powder/liquid ratio. Alongside, attention should be taken to the resultant mechanical properties of the set cement.

The design of the ZPC is actually multitasking work. Working and setting time should be controlled; the mechanical properties should be examined. Current trend in materials design is to consider sustainability. One of materials sustainability approaches is to utilize fewer materials as possible, simplifying the synthesis process and utilizing abundant materials, which is part of the vision for this research.

Experimental

The liquid part of the prepared cement consisted of diluted polyacrylic acid (Polysciences Inc., Germany). The molecular weight of the polyacrylic acid was 45,000 and the concentration was set to 44wt% via dilution with deionized water to prepare a solution with relative viscosity of about 1.2. Rotational viscometer (VR3000 MYR, Spain) was used to measure the viscosity of the prepared liquid.

The solid part of the prepared cements was consisted of two components. The first was the zinc oxide powder (DIDACTIC, Spain). The average particle size was measured via laser particle size analyzer (SHIMADZU SALD-2102, USA) and found 347 nm. The second component of the solid part of the cement was high silica glass. The high silica glass was prepared from waste soda lime glass and Urdhuma flint (Urdhuma location, Al-Ga'ara, depression western desert of Iraq). The soda lime glass was brought from Al-Taji Glass Manufacturing Site of the Ministry of Industry, Baghdad, Iraq. It was the remnants of the fluorescent lamp production process. The chemical analysis was provided by Al-Taji Glass Manufacturing Site as shown in table (1). The Urdhuma flint and its chemical analysis shown in table (1) were provided by the State Establishment of Geological Survey and Mining of Ministry of Industry and Minerals, Baghdad, Iraq.

To prepare high silica glass, the soda lime glass was crushed and ball milled to pass through 53µm sieve set. The Urdhuma flint powder also passes through similar sieve set. A 1kg batch of 70wt% of soda lime glass was mixed with 30wt% of Urdhuma flint in a porcelain jar laboratory blender for 1hr. The mix was then placed in an alumina crucible and fired for 1hr in 1000°C. The resultant glass was crushed and milled with alumina jar and balls for 30 min to improve homogeneity. The resultant powder was fired, crushed, and milled once more to produce the final high silica glass. The formulated high silica glass should contain not less than 80wt% SiO_2.

The glass was pulverized by blade miller (Bosch, Germany), and then fractionated through 150µm, 106µm, 75µm and 53µm sieve set. Further fractionation for the powder below 53µm was done via sedimentation method [17], during which, the powder particles were allowed to fall freely from deionized water suspension for a given sedimentation period followed by withdrawn of the upper 5cm of the suspension. The process was repeated several times until the upper 5cm of the suspension was almost clear after the sedimentation time. Two sedimentation times were employed (2.5 min, 15 min and 60 min) which result in approximate particle sizes of 25µm, 10µm, and 5µm respectively as measured via laser particle size analyzer.

The high silica glass was added to the zinc oxide powder in weight ratios of 5%, 10% and 15% for each particle size range mentioned above. The blend was mixed in rotated alumina jar for 90 min, where the axis of rotation was inclined by 30° above the horizontal axis. The zinc oxide-high silica glass blends and the 44wt% diluted polyacrylic acid were utilized to prepare the cements with powder/liquid ratios of 1.5:1 by weight. Working and setting times and the mechanical properties were evaluated as shown below.

The working and setting times of the cements were evaluated and guided by the procedure specified by the American Dental Association ADA [18], which is different from Gillmore Needles test as follows; Thermocouple type pt100 (accuracy ±0.1°C) was used to monitor the temperature of the cement mix. The cement was casted in (4mm diameter × 6mm height) cavity in polyethylene after 30 sec mixing time. The thermocouple tip was inserted to 1mm depth in the cement cast. The time at the max setting temperature was recorded as the setting time. As well, the halfway between the initial and the max temperature was recorded as the working temperature, which signifies the working time. The average of five measurements was accounted for each cement mix.

Compressive strength, flexural strength and flexural modulus, were examined in reference to ADA procedure [18]. The cements were mixed for 30 sec and casted for compressive strength measurements. The casting was in steel split mold with internal dimensions of 6 mm high and 4 mm diameter. As well, steel mold of internal dimensions of 25×2×2 mm was used to prepare cement casts for three-point loading measurements to determine flexural strength and flexural modulus. All casts were kept in their molds for 10 min in the environmental chamber at a temperature of 37°C and humidity of 50%. The chamber was made of Pyrex plates with suitable opening to place and withdraw the casts. The temperature was maintained via hotplate with temperature control and the humidity via heating up water in a glass beaker on the plate. Little experimentation was performed to maintain the target temperature and humidity. The temperature and humidity were read digitally via thermo-hygrometer (CHEERMAN KT-903, China). After that, the cast and their molds were placed in water path at 37°C for 60 min. Subsequently, the specimens were removed from the mold and were kept in the same water path for 24 hours.

The dimensions of the specimens were measured via micrometer after collecting from the water path. The compressive and flexural strengths were measured with a universal testing machine (TIME GROUP INC, China) system immediately. A compressive load was applied at a rate of 0.75 mm/min, along the long axis of the specimen for all measurements. The maximum force at failure was measured to calculate compressive strengths, also, the maximum force of failure was used in flexural strength determinations, while, the linear region of the stress-strain curves were used to determine the flexural modulus. Ten specimens were prepared for each measurement, and the standard deviations were calculated.

The infrared spectra for the soda lime glass, high silica glass and crushed ZPC (glass content 15wt%, 15µm) powders were measured using FTIR spectrometer (SHIMADZU, Kyoto - Japan). The powders were first mixed with KBr powder and pressed into disks before subjecting to FTIR measurements. Optical micrography (microscopes, Inc. USA) was performed to examine the microstructure at the fracture surface of the ZPC (glass content 15wt%, 15µm).

Results and Discussion

The first test was the apparent consistency of the cement when the solid part (nano sized ZnO+ 5, 10, 15 wt% high silica glass) was mixed with the liquid part (dilution of 44wt% of polyacrylic acid with deionized water). Incorporating high silica glasses with size ranges of

150μm, 106μm, 75μm, and 53μm results in cement mixes that did not show adequate apparent consistency. Thus, these size ranges were discarded. On the other hand, when the high silica glasses of average particle sizes of 25μm, 10μm and 5μm were included, the cements show obvious consistency.

The results of working times and setting times with respect of the particle size of high silica glass are shown in figures 1 and 2 respectively. The working time and setting time for cements with the solid part of ZnO with no glass addition is denoted as '0wt%'. These figures show two main features; the first is that both working and setting times were increased with higher content of the high silica glass. The second feature is that the lower particle size is more effective in increasing the working and setting times. Closer look at the figure tells that the addition of 5wt% and 10wt% of the glass led to an obvious increase in working and setting times. On the other hand, increasing the addition of the glass to 15wt% results in a minor increase the working and setting times compared with the glass addition of 10wt%. Furthermore, the smaller particle sizes of the glass additions were more effective in increasing working and setting times; yet, the results for 5μm are close to that of 10μm.

The standard deviation of the working and setting times measurements was about 1%. The results show that utilizing the glass powder of average particle size of 5μm, 10μm and content of 10wt%, 15wt% gives values of the working and setting times that are not significantly different. However, lower working and setting time were observed with the glass powder of average particle size of 15μm and/or content of 5wt%. This result makes a good space for the designer for cost considerations. In other words, the materials designer can choose the reduced cost options of glass powder of average particle size of 10μm rather than that of 5μm and content of 10wt% instead of 15wt%.

The infrared spectrums for the soda lime glass, the prepared high silica glasses and ZPC are presented at figure 3. The interval of 400-1000cm^{-1} is displayed; which is usually utilized to observe crystalline and amorphous silica. The peak position at 800 cm^{-1} shown in spectrums is common to quartz and vitreous silica; while the small peak at 462 cm^{-1} of the quartz cannot be distinguished from other small peak of 464 cm^{-1} of the vitreous silica. The absorption peaks dedicated to quartz at 695 and 514 cm^{-1} are not noticed on the spectrograms. Consequently, the peak at 800 cm^{-1} merely indicates amorphous silica for the three measured materials. In addition, the high similarity in the pattern of the high silica glass and the ZPC in the observed range of the spectrums indicate that the high silica glasses did not contribute to setting reaction of the prepared cements. This is different from the case of the glass ionomer cements that contain the calcium-aluminium-fluorosilicate-glass powders, where the glass contributes to the setting reaction via cross-linking with the acid through leached calcium and aluminium ions [1]. This, explains why the high silica glass was intentionally prepared for this work.

Accordingly, the high silica glass contribution to the prepared cements is expected to be the retardation of the cross-linking process. Consequently, the formation of the cross-linked chain was delayed which explains extending both the working and setting times. Thus, using cheap high silica glass material extends working time without considerably extending the setting time. This choice is better than adding other, more expensive, unsaturated carboxylic acids. Optical micrography, figure 4, revealed that the gel phase is dominant in the formation of the microstructure sufficiently to hide the remaining unreacted ZnO and the glass content at a magnification of ×800. This supports the view that the role of the glass particles in the gel matrix was merely intervening the cross linking process and delay it.

The results of compressive strength (standard deviation 2.5%) are presented in figures 5. A noticeable increase of the compressive strength was measured for the specimens when 5wt% of the glass is added which is a usual experience in powder technology. As well, more increase of the glass content turns out in more increase of the compressive strength but in smaller intervals. It seems that incorporating 15wt% of the glass is near a limiting value;

where more incorporates of glass particles may interfere with adhesion property of the gel matrix and affect the integrity of the cement. In addition, the results of the compressive strength in the case of incorporating the glasses of 5μm and 10μm are better than that of 15μm. This result may be attributed to that, it is difficult for the larger glass particles to rearrange their positions and to distribute the applied stress.

A different argument may be applied to explain the results of the flexural strengths (standard deviation 2%) shown in figure 6. As mentioned with the discussion of the working and setting times, the particles of the high silica glass may retard the cross-linking process. Accordingly, higher wt% of the glass particles and lower particle sizes that give in higher amounts of glass particles results in higher retardation of the cross-linking that enhance elastic properties and give higher flexural strengths. The same analysis was applied for the interpretation of the results for the flexural modulus; where again the higher wt% and lower particle sizes gave higher results. Increasing the wt% and decreasing the particle sizes of the high silica glasses in constant intervals turn out in increasing both the compressive and flexural strength in decreasing intervals. Hence, utilizing lower particle sizes and/or higher wt% of the glass possibly will not give extra benefit.

Once more, analysis of the mechanical properties elect the cements that incorporate glass powder of average particle size of 5μm or 10μm and content of 10wt% or 15wt% as seen for the results of the working and setting times. Thus, cost comprise may be included in the final selection of materials.

A further inspection of the results reveals an attractive behavior upon comparing the results of the mechanical properties with that of the working times. The increase of the setting times may indicate better final mechanical properties when utilizing the methodology of cement preparation of this work.

Conclusions

1- The setting and working times and the mechanical properties of the prepared zinc polycarboxylate cements are tailored utilizing high silica glass additions in the solid part of the cements.
2- Other additions than the high silica glass are not necessary in accordance with the preparation methodology of this work. Thus, adding other carboxylic acids to the liquid part of the cement was eliminated. Likewise, firing and grounding the zinc oxide powder to reduce its reactivity, and adding reinforcing fillers is no longer needed. This makes the production of the zinc polycarboxylate cements more echo and sustainable.
3- According to the preparation methodology in this work, the increase of the working times gives an indication to better mechanical properties of the cements.

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Table No. (1): Chemical analysis of Al-Taji Soda Lime Glass (SLG) and Urdhuma Flint (UFlint)

	SiO_2	Al_2O_3	Fe_2O_3	TiO_2	CaO	MgO	Na_2O	K_2O	SO_3	Cl	L.O.I.
SLG	72.29	1.45	0.6	0.01	5.0	3.45	16.4	0.35	0.15	0.05	–
UFlint	98.4	0.4	0.05	–	0.3	0.3	–	–	–	–	0.55

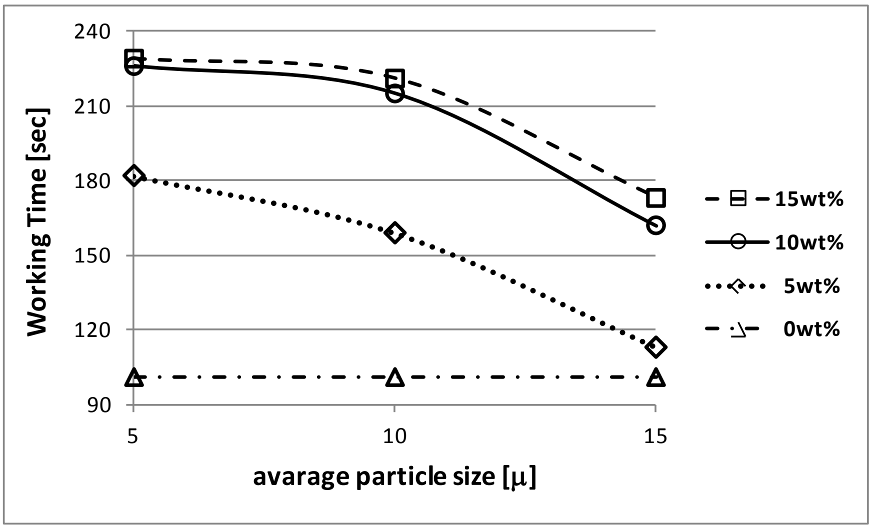

Figure No. (1): Working times for the prapared cements for different average particle sizes of the added high silica glass to ZnO powder

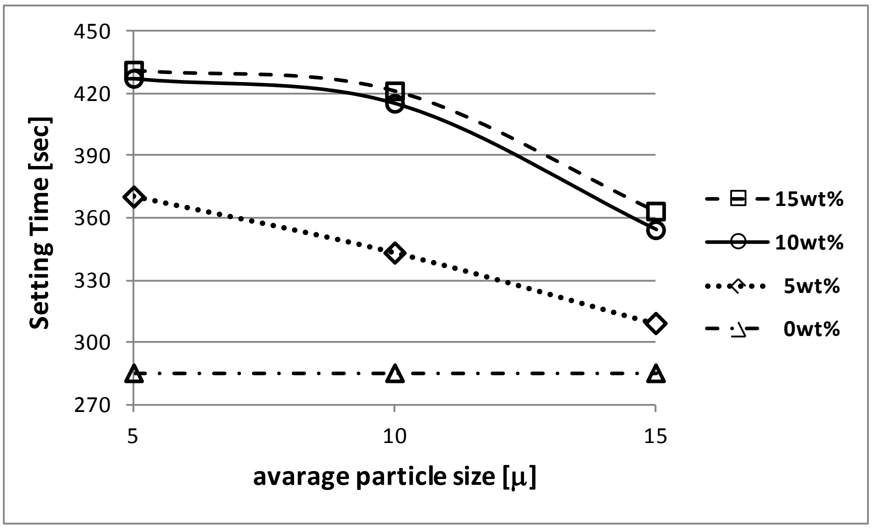

Figure No.(2): Setting times for the prapared cements for different average particle sizes of the added high silica glass to ZnO powder

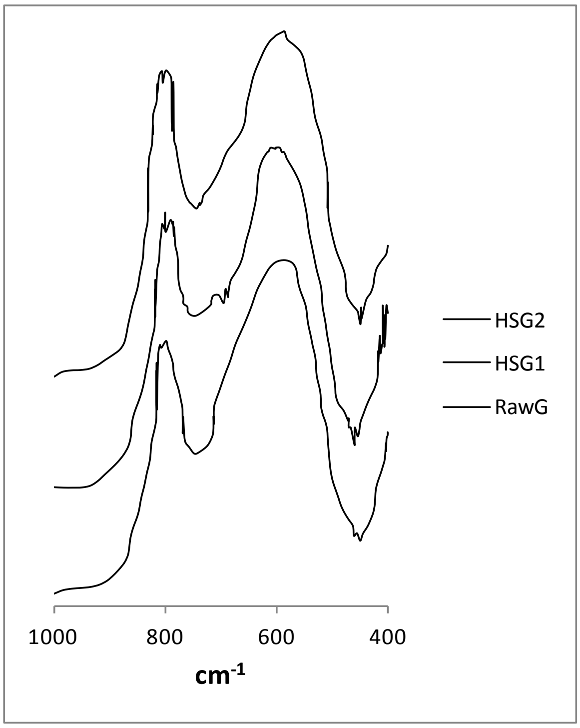

Figure No.(3): FTIR spectra for the soda lime glass (RawG), High Silica Glass HSG1 and the Zinc Polycarboxylate Cement HSG2 (high silica glass content 15wt%, 15µm)

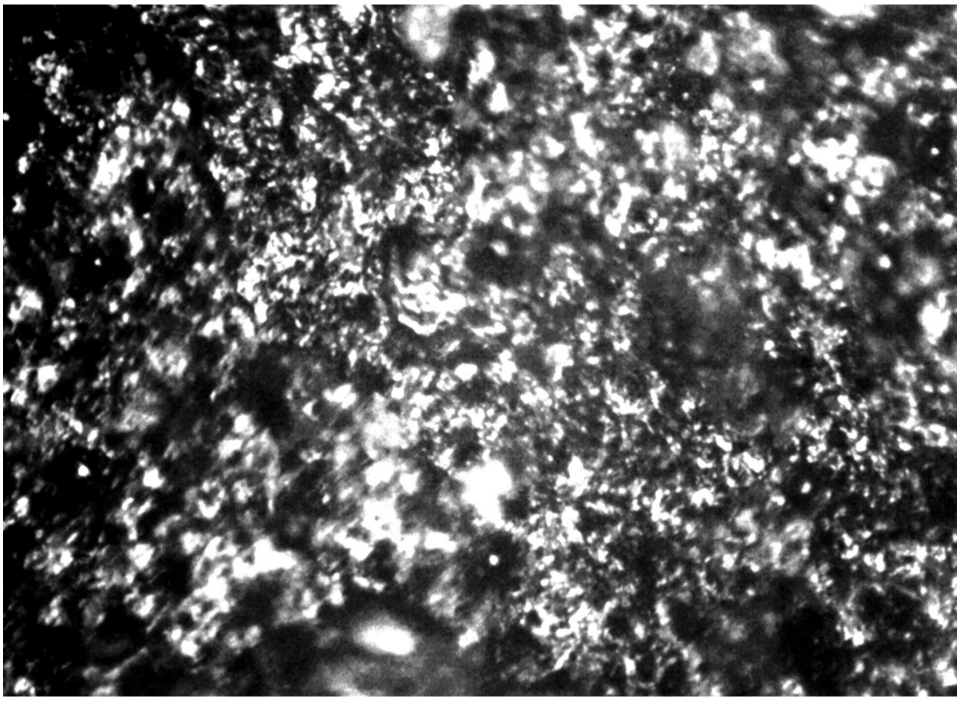

Figure No.(4): Optical micrograph ×800 for a fracture surface of the prepared Zinc Polycarboxylate Cement. The solid part mixed with 15wt%, glass of 15µm average particle size

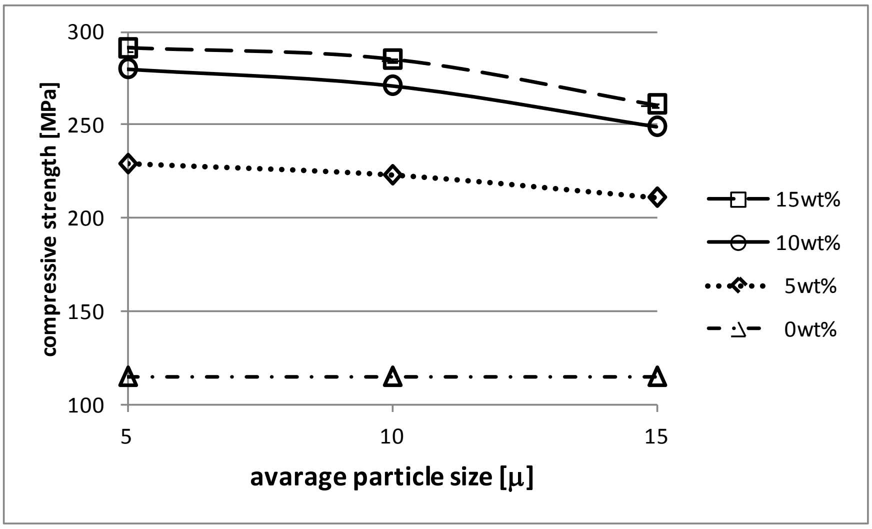

Figure No.(5): compressive strength for the prapared cements for different average particle sizes of the added high silica glass to ZnO powder

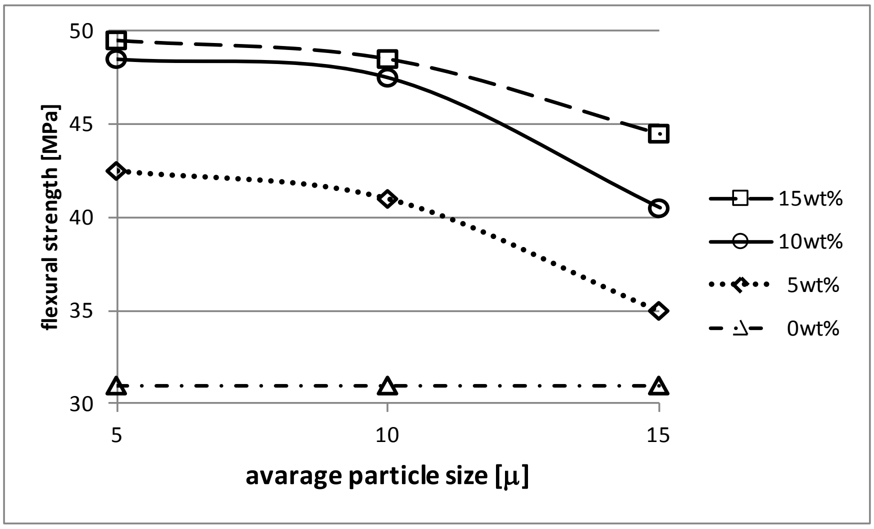

Figure No.(6): flexural strength for the prapared cements for different average particle sizes of the added high silica glass to ZnO powder

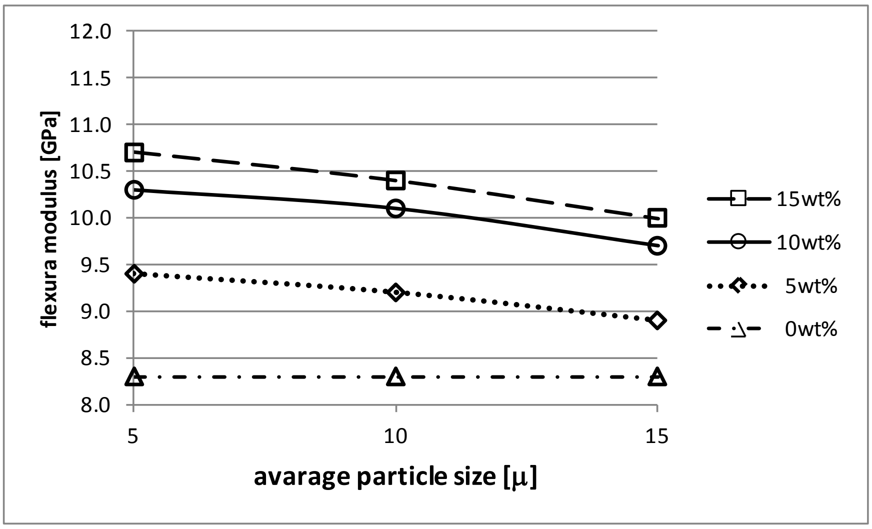

Figure No.(7): flexural modulus for the prapared cements for different average particle sizes of the added high silica glass to ZnO powder

إعادة تصميم سمنت الأسنان نوع زنك-بوليكربوكسيل

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استلم البحث:8 تشرين الاول 2013 قبل البحث في4 كانون الاول 2013

الخلاصة

تمت مراجعة تركيبات وخواص سمنت الأسنان نوع زنك-بوليكربوكسيل ثم حضرت مجموعة جديدة من هذا النوع من السمنت من خلال تعديل الجزء الصلب من السمنت. وقد حضر زجاج عالي السيليكا باحجام دقائق مختلفة واضافته بنسب وزنية مختلفة الى اوكسيد الزنك. ويتكون الجزء السائل للسمنت المحضر فقط من حامض البولي-اكريلك بتركيز 44%. ولذلك، تم حذفت الاضافات المعتادة والغالية الثمن للجزء السائل والجزء الصلب من السمنت. قيس زمن التحضير، ووقت التصلب، و تحمل الانضغاط ، وتحمل، ومعامل الانحناء تبعا لمواصفات الجمعية الأمريكية لطب الأسنان ADA. تتصف مجموعة السمنت المحضرة باطالة زمن التحضير من دون زيادة كبيرة في وقت التصلب. وكذلك، تحسنت المواصفات الميكانيكية بشكل واضح مقارنة بالسمنت الاصلي.

الكلمات المفتاحية: سمنت الزنك بوليكربوكسيل، زمن التحضير ووقت التصلب، تحمل الانضغاط، تحمل ومعامل الانحناء