The effect of incorporation of prepared Ag-Zn Zeolite on some properties of heat polymerized acrylic denture base materials

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ABSTRACT

Background: Poly (methylmethacrylate) is the most widely used material in denture fabrication. The characteristics of acrylic resin which support microorganism development can threaten the oral health of denture users. This study was assigned to prepareand incorporate Ag-Zn zeolite powder into heat cured denture base material as antimicrobial material and to investigate its effect on some properties of heat cured acrylic denture base materials.

Materials and methods: Sliver –zinc zeolite was prepared by ion exchange method and characterized then incorporated into poly (methylmethacrylate) powder in0.5% by weight. Specimens were constructed and divided into 6 groups according to the using tests; each group was subdivided into 2 groups. The tests conducted in this study were: impact strength test, transverse strength test, surface hardness test, surface roughness test, water sorption test, water solubility test and color change measurement after addition. The results were statistically analyzed by t-test at p value≤ 0.05.

Results: Characterization methods results showed the incorporation of sliver and zinc ion without change of zeolite structure framework. A non-significant effect resulted from the addition of 0.5% sliver –zinc zeolite on the impact strength, transverse strength, surface hardness, surface roughness and cause no change in color of heat cure denture base. Also a highly significant decrease in water sorption and a significant increase of water solubility were observed.

Conclusion: Preparation of sliver-zinc zeolite could be performed successfully and the addition of 0.5% of antimicrobial sliver-zinc zeolite into heat cure acrylic had a non significant effect on the impact strength, transverse strength, surface hardness, surface roughness and did not change the color, also there was a significant decrease in water sorption and increase in water solubility of acrylic resin.

Key words: Denture stomatitis, Antimicrobial agent, Ag- Zn Zeolite. (J Bagh Coll Dentistry 2015; 27(1):63-69).

INTRODUCTION

It is well-known that removable denture bases fabricated from heat-polymerized acrylic resins may act as a reservoir for microorganisms and contribute to re-infection in denture wearers. Biofilm deposition on the surface of acrylic denture bases is enhanced by the characteristics of the material, especially its porosity, irregularity and absorption [(1)]. Systemic or local antimicrobial agents have been prescribed for eliminating the fungal population; however with microbial resistance, bad healthcare and cost, research on antimicrobial denture base materials is needed for its prevention and care [(2)].The mechanism of zeolite's antimicrobial effect is based upon ion exchange reaction of the antimicrobial cations within which are present zeolite pores [(3)].

It has been reported that silver ion exchanged zeolites have good antibacterial activity and therefore have a potential in the medical field to enhance antimicrobial properties of polymers [(4)].

Since zeolite is a natural mineral, non-toxic, non-carcinogenic and has a high importance as food supplement and medical treatment agent for both humans and animals, it is completely safe to be used in medical devices [(5)].

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It is important to evaluate the mechanical properties of acrylic resins containing zeolites because removable and complete dentures are subjected to repeated forces [(6)].

Nevertheless, the addition of small percentages of zeolite to poly (methylmethacrylate) may be effective against microorganisms; therefore its effect on mechanical properties may be less significant than the potential benefits, especially for patients who do not follow an adequate denture cleaning protocol [(7)].

MATERIALS AND METHODS

Ag-Zn zeolite can beprepared by ion-exchange method in water phase [(8,9)] Ion-exchange is obtained by contacting a 200g of zeolite type 13X with aqueous solution containing 10 g of sliver acetate and 100 g of zinc acetate; put it in a thermostat shaker with 80 rpm at 25°C for two hours.

Then the Ag-Zn zeolite rods were ground by planter ball mill and further characterized by Atomic Absorption spectroscopy (AAS), Infrared spectroscopy and X-Ray powder diffraction.

One hundred twenty acrylic specimens were prepared in this study using three different metal patterns which constructed by cutting stainless steel plates into desired shapes and dimensions using turning machine these patterns were used in the mold preparation by conventional flasking

technique using heat cure acrylic resin (SUPER ACRYL ®PLUS).The samples were divided into six groups according to the using tests and each group subdivided into two subgroups. The required weight of polymer powder and Ag-Zn Zeolite was weighed using digital electronic balance for both groups. Ag-zinc zeolite powder was added to polymer powder and mixed manually using mortar and pestle.

Mechanical and physical tests:

Impactstrength test:

The specimens were prepared with dimension (80mm ×10 mm× 4mm±0.2mm) (10) for unnotched specimens. Specimens were stored in distilled water at 37°C for 48 hour before the test [11]. The impact strength test was evaluated following the procedure recommended by the ISO 179 with charpy type impact testing device.

The specimens were supported horizontally at each end and struck by free swinging pendulum of 2 Joules released from fixed height in the middle. The scale reading gives the impact energy in Joules that absorbed to fracture the specimen when struck by a sudden blow.

The charpy impact strength of unnotched specimens were calculated in kilo Joules per square meter by the following equation:

$$\text{Impact strength} = \frac{A}{X.Y} \times 10^3 \ (Kj/M^2)$$

A: The impact energyabsorbedin Joules

X: Is the width of the specimens in millimeters

Y : Is the depth of the specimens in millimeters.[10]

Transverse strength test

Specimens were prepared with dimension (65mm×10±0.03mm×2.5±0.03mm) according to [11] Specimens were stored in distilled water at 37°C for 48 hour before the test [11].

The test was performed using Instron testing machine ,each specimen was positioned on bending fixture, consisting of 2 parallel supports (50)mm apart , the full scale load was 50 Kg, and the load was applied with cross head speed of 1mm/min by rod placed centrally between the supports making the deflection until the fracture occurred.

The transverse strength was calculated using the following formula:

$$S = \frac{3PI}{2bd^2}$$

S= Transverse strength (N/ mm^2)

P= maximum force exerted on specimens (N)

l=the supporting width in mm=50

b= width of the samples (mm)

d= depth of the samples (mm) [12]

Surface roughness tests:

Specimens with dimension of (65 mm×10 mm×2.5 mm)were prepared All specimens were immersed in distilled water at 37°C for 48 hours before being tested [11].

The profilometer device (surface roughness tester) was used to study the effect of Ag-Zn zeolite on the micro geometry of the test surface and this device has surface analyzer to trace the profile of the surface irregularities.

The profilometer records by its scale all the peaks and recesses which characterized the surface of the acrylic specimens, which were placed at stable bench. Three different location were selected for each specimen (the same for all specimens) then the analyzer pass along the specimen surface for 11 mm distance then the mean of three readings were recorded for each specimen.

Surface Hardness test:

Specimens of heat cure acrylic resin were prepared with a dimension (65mm ×10 mm× 2.5 mm±0.2mm).All specimens were left in distilled water at37°C48 hours until tested [11].

Test was performed using durometer hardness tester (shore D hardness) that was fabricated according to American National Standard/ American Dental Association [13] which is suitable for acrylic resin material. The instrument consisted of blunt-pointed indenter 0.8mm in diameter that tapered to a cylinder 1.6mm.The indenter is attached to a digital scale that is graduated from 0 to 100 units. The usual method is to press down firmly and quickly on the indenter and record five maximum reading as shore hardness, measurement were taken directly from the digital scale reading. Five measurements were done on different areas of each specimen (the same selected area of each specimen and the average of the five readings was calculated.

Water sorption and solubility test:

Acrylic disc specimens were prepared by using metal pattern with a dimension of (50 mm ± 1 mm in diameter and 0.5 mm ± 0.1 mm in thickness). [11].

The specimens were dried in desiccators containing freshly dried silica gel. The desiccator was stored in an incubator at37°C ± 2°C for 24 hours after that the specimens were removed to room temperature for one hour then weighed with a digital balance with an accuracy of (0.0001g). This cycle was repeated until a constant mass (M1) "conditioned mass" was reached which

indicated that the weight loss from each disc was not more than 0.5mg in 24 hours [11]

The two groups reached to Ml after 4 days, then all discs of both groups were immersed in distilled water for 7 days at 37°C ± 2°C [11]. The discs were removed from the water with a dental tweezers wiped with a clean dry towel until free from visible moisture and weighed one minute after removal from the water; this mass was recorded as (M2).

The value of water sorption were calculated for each disc from the following equation: [11]

$$WSP = \frac{M2 - M1}{S}$$

WSP: Water sorption in mg/cm^2

$M2$: The mass of the disc after immersion in distilled water (mg)

$M1$: The mass of the disc before immersion in distilled water (conditioned mass) (mg)

S: Surface area of the disc (cm^2)

In order to obtain the value of solubility the discs were again reconditioned to a constant mass in the desiccators at 37°C ±2°C and weighed every 24 hrs as done in the first time for sorption test and the reconditioned mass was recorded as($M3$). The whole group was reached to M3 within 4 days.

The solubility was determined for each disc by the following equation:

$$WSL = \frac{M1 - M3}{S} \quad (11)$$

WSL: Solubility (mg/cm^2)

$M1$: The conditioned mass (mg)

$M3$: The reconditioned mass (mg)

S: The surface area of the disc (cm^2)

Color change measurement:

A disc with same dimension and processing procedure that used for water sorption and solubility test were used for color change measurement.

The color change was measured by the objective method (spectroscopic study) using spectrophotometer device by measuring the amount of absorbed light in nm.

RESULTS

The results were obtained from 120 experimental specimens of acrylic resin classified into:

- Control group: no Ag-Zn zeolite added.
- Experimental group: 0.5% concentration of Ag-Zn zeolite.

Results of the conducted tests includes:-

A- Characteristic methods.

B- Mechanical and physical test.

The FTIR spectra for pure and Ag-Zn zeolite showed the characteristic peaks of zeolite material. At the range of (400-4000cm^{-1}) fig.(1), both spectra of pure and Ag-Znzeolite were quite similar except shift of O-H and T-O stretching vibration observed in the spectrum of Ag-Zn zeolite.

At the range of (200-400cm^{-1}) fig.(2) ,there was appearance of new peak at 217.94cm^{-1}and 243.01cm^{-1}.A peak splitting at 325.95cm^{-1} into 327.88cm^{-1} and 322.09cm^{-1}.A different peak intensity showed at 279.66cm^{-1} for the spectrum of Ag- Zn zeolite; otherwise both spectra were the same. For the spectrum of PMMA-Zeolite composite fig. (3), was quite similar to that of the pure PMMA except for the appearance of 2 new peaks at 1645.17cm^{-1}-1390.58cm^{-1} and reduced intensities for the following peaks(1267.14cm^{-1}, 1242.07cm^{-1}, 1066.56cm^{-1}, 989.41cm^{-1}, 838.98cm^{-1}, 754.12cm^{-1}).

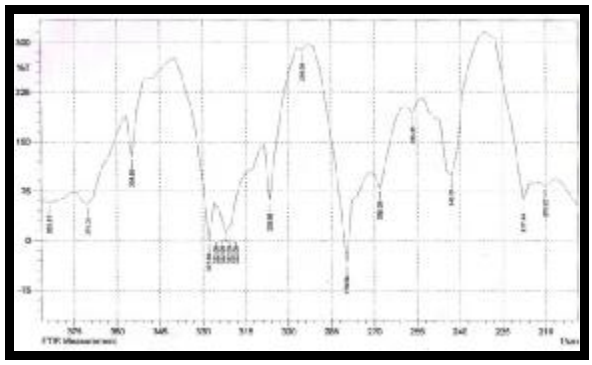

Figure 1: FTIR spectrum atthe range of (200-400cm^{-1}) of Ag-Zn zeolite

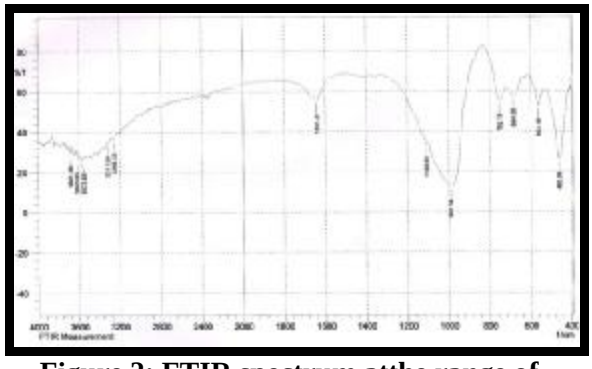

Figure 2: FTIR spectrum atthe range of (400-4000cm^{-1}) Ag-Zn zeolite

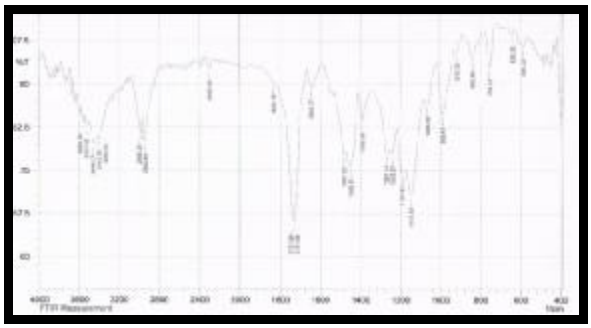

Figure 3: FTIR spectrum of PMMA/Ag-Zn zeolite composite

XRD patterns for both pure and Ag- Zn zeolite showed that the relative intensity of the characteristic peaks changed without any significant shift of the respective peak position. The chemical analysis carried out by Atomic Absorption Spectroscopy showed the following composition 5%, 2% and 2.2% for Sodium, sliver and zinc ions. While for particle size measurement, Laser diffraction particle size analyzer results showed it in the range (0.5-1µm).

Impact strength test

The results of the impact strength for heat cured acrylic resins specimens of the control group and the experimental group showed mean values of 8.68 and 8.43 KJ/M^2, respectively.

t-test was used to examine the effect of Ag- Zn zeolite addition to PMMA on the impact strength of acrylic resin. The result revealed a non significant difference between both group at p value ≤0.05.

Table 1: Descriptive data and t-test of impact strength test results in KJ/M^2

Materials	Descriptive Statistics and t-test				
	Mean	SD	t-test	p-value	Sig.
Control	8.68	0.17	1.25	0.227	(NS)
Experimental	8.43	0.60			

NS= Non-Significant

Transverse strength test

From the table below, the mean value of transverse strength for the control group is 51.22N/mm² while for the experimental group is 50.90N/mm². t-test showed a non significant difference between both group at p value ≤0.05.

Table 2: Descriptive data and t-testof transverse strength test results in N/mm²

Materials	Descriptive Statistics and t-test				
	Mean	SD	t-test	p-value	Sig.
Control	51.22	2.56	0.27	0.788	(NS)
Experimental	50.90	2.67			

NS= Non-Significant

Surface hardness test

The samples of the control group showed a lower mean value of the surface hardness test which is equal to (84.19).although the experimental group showed a higher mean value than the control group which is equal to (85.37), t-test result revealed a non significant difference between both groups at p value ≤0.05.

Table 3: Descriptive data and t-test of surface hardness test results

Materials	Descriptive Statistics				
	Mean	SD	t-test	p-value	Sig.
Control	84.19	1.51	-1.46	0.163	(NS)
Experimental	85.37	2.06			

NS= Non-Significant

Surface roughness test

The mean value of the surface roughness test for control group was higher than that of the experimental group which are equal to (1.81, 1.73) respectively. t-test showed a non-significant difference between both groups

Table 4: Descriptive data and t-test of surface roughness test results

Materials	Descriptive Statistics and t-test				
	Mean	SD	t-test	p-value	Sig.
Control	1.81	0.22	0.59	0.562	(NS)
Experimental	1.73	0.36			

NS= Non-Significant

Water sorption test

A higher mean value was for the control group (0.3mg/mm²) while for the experimental group was (0.28mg/mm²). t-test was highly significant with p value of (0.000) suggested a highly significant statistical difference between both group.

Table 5: Descriptive data and t-testwater sorption test results (mg/cm²)

Materials	Descriptive Statistics and t-test				
	Mean	SD	t-test	p-value	Sig.
Control	0.30	0.01	4.64	0.000	(HS)
Experimental	0.28	0.01			

HS= Highly Significant

Water solubility test

The mean value of the experimental group was higher than that of the control group which equal to (0.06, 0.03 mg/cm²) respectively. t-test result revealed a significant difference between both group at p value ≤0.05.

Table 6: Descriptive data and t-test of water solubility test results(mg/cm²)

Materials	Descriptive Statisticsand t-test				
	Mean	SD	t-test	p-value	Sig.
Control	0.03	0.02	-2.20	0.041	(S)
Experimental	0.06	0.04			

S = Significant

Color change measurement

The mean value for the experimental group and the control group were equal to (1.18, 1.14) respectively. Statistically a non significant difference between both groups according to t-test result at p value ≤0.05.

Table 7: Descriptive data and t-test of Color change measurement results

Materials	Descriptive Statistics and t-test				
	Mean	SD	t-test	p-value	Sig.
Control	1.14	0.04	-1.04	0.312	(NS)
Experimental	1.18	0.10			

NS=Non-Significant

DISCUSSION

Preparation, addition and characterization of Ag-Zn zeolite:

This study selected the preparation and the use of zeolite as a vehicle for antimicrobial cations such as sliver and zinc ion due to its sorption and ion exchange properties. This in agreement with Orha *et al.* [14]

Sliver and zinc ion were used as the cations of choice to be used in denture base material because they possess strong antibacterial and antifungal activity, this in agreement with Abe *et al.*[15] that assessed the incorporation of sliver- zinc zeolite into tissue conditioner to improve the antimicrobial property.

The best concentration of Ag-Zn zeolite was 0.5%which showed the least and negligible adverse effect on acrylic resin properties in the same time it had antifungal properties as found by Mutneja *et al.*[16]

The result of AAS showed comparable ion concentration was obtained for both sliver and zinc ion in order to obtain the same antimicrobial effectiveness of both ions. This agreed with Kaali *et al.*[17].

The results of FTIR for zeolite before and after ion exchange showed some changes due to the ion exchange. The change of vibration bands of T-O asymmetrical and symmetrical stretching in the range of (400-4000cm^{-1}) also in the range of (200-400cm^{-1}) the changes and the appearance of new peaks all indicate the incorporation of sliver and zinc ion into zeolite. This in agreement with Orha *et al.* [14].

The FTIR spectrum of PMMA- Ag-Zn zeolite, recorded the changes as the appearance of the vibration band at 1639.33cm^{-1}and the disappearance of the vibration band at 979-1200cm^{-1} which belong to the zeolite spectrum could be due to the interaction between PMMA and the Ag-Zn zeolite and this could explain that some mechanical properties of PMMA such as impact and transverse strength, surface hardness and roughness weren't changed, also it could explained the reduced in water sorption test**.**

The XRD patterns of both pure zeolite and modified zeolite were almost similar; also no crystalline pattern was observed for Ag and Zn ion that could be because their fine distribution in zeolite lattice indicating that the incorporation of Ag and Zn had little effect on crystalline structure of host zeolite.

These results comparable were with Zendehdel *et al.*[18] Also in agreement with Orha *et al.*[14].

Impact strength test

The results of impact strength test showed that the addition of 0.5% of Ag-Zn zeolite powder to heat cure acrylic resin had a non significant effect on the impact strength .It may be due to the small particle size in range between (0.5-1µm) and small percentage of zeolite added.

These results were explained by that adding sliver-zeolite to heat cure resin tend to decrease the material property depending on the additive concentration of the antimicrobial zeolite [19]. Also in agreement with Casemiro *et al*, [6] and coincided with Hassan *et al.*, [20].

Transverse strength test

The current study showed that the addition of the 0.5% of Ag-Zn zeolite causes a non significant change in the transverse strength values.

Nakanoda *et al.*, [19] found that small amount of antimicrobial zeolite added to acrylic resin had less effect on material property.

This disagree with the result of with Casemiro *et al.*, [6] and Mini *et al.*, [21]who found a significant decrease of flexural strength in comparison to the control groups was observed with the addition of 2.5% of zeolite. This can be explained by high percentage of the added zeolite in comparison to the current study.

Surface hardness test

Although the mean value of the surface hardness test for the experimental group was higher than that of the control group but it was statistically insignificant under the explanation that zeolite mainly composed of silica with small particle size and high surface area leading to better interfacial adhesion of the composite material [22].

This was in similarity Alnamel, [23] who found that the increase in surface hardness of acrylic resin after the addition of silicon dioxide may be attributed to the randomly distributed particles of a hard material in acrylic matrix.

Surface roughness test

Results showed that both experimental and control group had comparable mean values of surface roughness test which was statistically insignificant.

This could be due to small percentage of the Ag-Zn zeolite with small particle size and well dispersion so few particles would be involved with the surface of the specimens, also the profilometer which was used in measuring the surface roughness was concerned with the outer surface of the composite specimen and not with the inner surface that's why the surface roughness of the experimental group was not increased. This result was comparable with both Alnamel [23] and Noori [24].

Water sorption test

From the result of this study there was a highly significant difference between the mean values of both groups as the experimental group showed a lower mean value than the control group.

As acrylic resins absorbed water slowly over a period of time, primarily because of the polar properties of the resin molecules and by the addition of filler particles, the actual number of PMMA molecules available on the surface of the specimen for water sorption to occur decreased as compared to the control group [25, 26]

The results also in agreement with Kaali *et al.* [17] although zeolite is a hygroscopic material and its content in the composite may result in an increase in water uptake but this hygroscopic behavior of zeolite depend on its amount in the composite as the zeolite content in the composite. Also they found that the different ion within zeolite framework may influence zeolite hygroscopic behavior and water absorption as well.

Water solubility test

The result of this test showed a significant increase in water solubility of the experimental group in comparison to the control group.

This could be due to the ion exchange property of zeolite for the antimicrobial cations within its lattice with the surrounding environment. This needs further investigation and analysis for the released ions.

This comparable with Kaali *et al.* [17] who found through a SEM examination of the surface of Ag-ZN -Cu exchanged zeolite /poly urethane composite after in vitro degradation that surface alteration due to ion activity and the capability to diffuse to the surface of polymer and migrate out in order to form equilibrium between the bulk and the environment, lattice with the surrounding environment. This needs further investigation and analysis for the released ions.

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Color change measurement using spectrophotometer

In the present study the color of PMMA before (control group) and after (the experimental group) the addition of Ag-Zn zeolite was measured using spectrophotometer device [27].

The results of the color test showed that there was a non-significant difference in light absorption between both groups.

This result may be duo to small percentage of zeolite added.[6,17]

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