

## Shear Bond Strength of Acrylic Soft Liner after Incorporation of Silver- Zinc Zeolite

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Received June 24, 2019.

Accepted for publication July 16, 2019.

Published September 2, 2019.

### Abstract

**Background** The main disadvantages the soft liners, is the absence of a durable bond to the denture base, this caused adhesion failure between them, creating an environment for colonization bacterial and compromise the durability of the Soft denture liners. The objective of this study was to evaluate the effect of silver-zinc zeolite incorporated into heat cure acrylic soft liner on shear bond strength.

**Materials and methods** Thirty specimens of heat cure soft liner were prepared and divided into three groups after incorporation of Ag-Zn as follows; Control Group: which have 10 specimens without Ag-Zn zeolite. Experimental Group: which have ten specimens with 0.5% by weight of Ag-Zn; Experimental Group: which have 10 specimens with 0.75% weight of Ag-Zn zeolite plastic pattern sample which used for acrylic block fabrication were (75 mm length × 25 mm width × 5 mm depth. The handle of acrylic block thickness is 13mm. Shear bond strength of all specimens was evaluated. **Results** The LSD test showed The experimental groups of 0.75% and 0.5% significant difference compared with the control group ( $p < 0.05$ ). While the comparison between experimental groups (0.75 % and 0.5 %) had recorded non-significant difference at  $p > 0.05$ . **Conclusions** incorporation from (0.5% and 0.75%) of Ag-Zn Zeolite into heat soft liner had a significant effect on shear bond strength.

**Keywords: Ag-Zn Zeolite, soft liner, shear bond strength, mode of failure.**

### Introduction

Soft denture lining materials show a very significant role in removable prosthodontics. Soft liner materials are utilized to provide a cushioning influence for maintaining the health of deformed, traumatized, swollen mucosa, by absorption and identical redistribution of stresses over the whole area covered by a denture; hence the distortion of the oral mucosa will be eliminated. Long-term soft denture lining materials that can continue in the oral cavity for at least four weeks, several months, even years. The use of long-term soft denture lining materials is recommended in edentulous patients with sharp, atrophied alveolar ridges, patients had thin atrophic mucosa, mucosa presents insufficient tolerance to the load transmitted by the dentures, the dentures displays poor retention, patient experience pain at nerve ending locations, for relining in implantology and for performing postoperative obturation (El-Hadary et al, 2000, Bulad, 2004). The most commonly used Long-term soft denture

lining materials are plasticized acrylic resin available in liquid and powder. The plasticizer is responsible for maintaining the material softness therefore, their use extended for several months and years (Pinto et al, 2002; Chladek et al, 2014). These materials have some difficulties related to their use. The main serious problem with soft denture liners is the failure of the bond between the denture base and the soft denture liner (Alaa, 2013). Easily colonized these materials and infected by *Candida* species and bacteria leading to denture-induced stomatitis. Antimicrobial zeolites have been incorporated. The evidence suggests that silver zeolite is a potentially effective antimicrobial agent. The studies had shown favorable long-term of the soft liner had antimicrobial effects containing silver zeolite on *Candida albicans* and the bacteria *Staphylococcus*. (Singh et al, 2018). Zeolite is a crystalline structure consist of aluminosilicate with its porous structure that allowed various ions like zinc, silver was embedded within pores. (Samiei et al, 2017) Antimicrobial zeolites have been utilized as filler with dental materials to inhibit or decrease contamination bacterial, fungal, and to assess some mechanical properties (Casemiro et al, 2008). The shear bond test is considered a beneficial technique to assess the bond strength because it more associated with clinical settings than the tensile test. But, the stresses in soft linings are distributed unevenly and focused near the edges (Chladek et al, 2014). The present study aims to evaluate the incorporation of 0.5%, 0.75% of Ag-Zn Zeolite by weight into heat cure acrylic soft liner on shear bond strength.

## Materials and methods

### Preparation of silver-zinc zeolite

Amount of each compound (silver acetate (Ag), Zinc acetate (Zn), Zeolite 13X Fluka, swiss) as shown in( Figure1-A), was determined approximately according to the molar mass of each compound, antimicrobial Ag-Zn zeolite can be prepared by an ion-exchange technique in the water phase (Azzez and Fatah, 2015). Ion-exchange is achieved by the contact between a quantity from zeolite and quantity of solution, for a period of time range from 2 hours to 48 hours at a constant temperature, separating the two components of the exchanging ions (Shery, 2003). A 25gm of Zn<sup>2+</sup> acetate was added to 500ml of highly deionized water in a conical flask. Magnetic mixing capsule was used inside the conical flask, and then placed it on the magnetic mixing device. A 2.5gm of Ag<sup>+</sup> acetate was added to the conical flask after dissolved Zn<sup>2+</sup> in the deionized water, and then covered with aluminum foil in order to prevent light exposure for the solution. 50 gm of zeolite rods were added to the aqueous solution (metal ion salt) then, set it in a digital shaker thermostat with eighty round per minute at 25°C for 2 hours (Azzez and Fatah, 2015) as shown in Figure (1-B). The mixture was filtered by vacuumed filtration as shown in Figure (1-C). The Ag-Zn Zeolite rods should be dry in a vacuumed oven at 65°C overnight. Then, crushed to the powder by Planetary Ball Mill in Figure (1D). The average particle size (0.5-1 $\mu$ ) measured by Laser Diffraction particle size analyzer.

### Ag- Zn Zeolite characterization

#### A-Atomic Absorption Spectroscopy (AAS)

The AAS (Novaa, Germany) is an analytical method used to determine qualitative and quantitative of the elements present in a different sample. (García & Báez, 2012). By taken amount from the powder of Ag-Zn Zeolite between (0.05 - 0.1 gm). addition of (HNO<sub>3</sub>+HCL) dissolved solution and heating at (250 C). Taken the amount of dissolved solution (0.1 ml ) that equivalent (100) placed in volume flask (10) ml

that given mitigation (100)once. Insert wavelength (213) of  $Zn^{2+}$ , and then insert standard concentration for  $Zn^{2+}$  (0.1,0.2,0.4,0.8) ppm, this lead to appear absorption (Abs) for each concentration, and obtained a standard curve. The capillary tube of a device placed in a diluted solution of Ag-Zn powder. The device given amount of  $Zn^{2+}$  in a diluted solution finally inserted the amount of Zn in the special equation. The same way used to determine amount absorption of silver ( $Ag^+$ ) with standard concentration (1,2,4) and wavelength 328for  $Ag^+$ .

Concentration from device x volume flask x number diluted

Weight



B- X-ray fluorescence analysis (XRF, Oxford instrument, United Kingdom)

The XR used to determine the chemical composition of zeolite samples (Aisiyah et al, 2018). Taken amount (1gm) from powder of zeolite and press until it became like plate then placed inside the device.

C- Fourier transforms Infrared spectroscopy (FTIR Bruker, Germany)

The control and experimental specimens were tested by FTIR analysis. The FTIR used to determine any chemical reaction between acrylic soft liner and Ag-Zn Zeolite then, compare the results with the control by scrapping a small amount from the acrylic soft liner specimen (control and experiment) and mixing with potassium bromide (KBR) salt, pressing as a disk by using a mini hand press, (KBR) salt was used for aiding in transmission of IR rays through the specimen. FTIR spectra were obtained by placing the specimens to be analyzed on a specified position inside the FTIR ana

lyzer (Mustafa and Hamad, 2015).

### **Samples grouping**

Thirty specimens of heat acrylic cure soft liner were prepared and divided into three groups as follows;

Control Group: 10 samples without of Ag-Zn Zeolite incorporation

Experimental Group: 10 samples with 0.5% by weight of Ag-Zn zeolite.

Experimental Group: 10 samples with 0.75% by weight of Ag-Zn zeolite.

### **Incorporation of Ag-Zn zeolite in heat cures acrylic soft liner**

The Silver-Zinc zeolite powder was incorporated in the pre-defined percentages (0.5% , 0.75% by weight) into the polymer powder (Vertex). (Casemiro et al, 2008; Azzez and Fata, 2015). As follows:

1.Control Group:10g of powder, 4.4 ml of liquid

2.Ag -Zn Zeolite 0.5 % group: Ag-Zn zeolite (0.5 g), powder (9.5g), liquid (4.4 ml)

3.Ag -Zn Zeolite 0.75 % group: Ag-Zn zeolite (0.75g), powder (9.25g), liquid (4.4 ml)

### **Specimens design**

The plastic plate cut into suitable shape using a turning machine. For the evaluation of the shear bond strength between the acrylic soft liner and the acrylic denture base, the dimensions of plastic pattern which used for acrylic blocks fabrication were (75mm length × 25mm width × 5 mm depth. The thickness of the handle of the acrylic block is (13 mm).as in Figure 2-A).

### **Proportioning and Mixing a of acrylic soft liner and heat cure acrylic**

In the current research, 2.2g of powder and 1ml of liquid monomer were mixed according to the manufacturer's instructions. (Issa and Abdul-Fattah, 2015). Finishing was done by using the stone, acrylic burs followed by sandpapers, and then polishing was done as a conventional method. All specimens were stored in distilled water at (37°C) for 2 days according to ADA specification No 12 (1999). One of the heat cure acrylic block was placed over the other block leaving a space between them with dimensions (25mm length × 25mm width × 3mm depth, with a stopper of depth about 3mm for relining material application. (Mohammed et al, 2016; Yasser and Fata, 2017) Figure (2-B) which filled with wax, after wax elimination, fills the space of stopper with soft liner material (Yasser and Fatah, 2017).

### **Testing procedure**

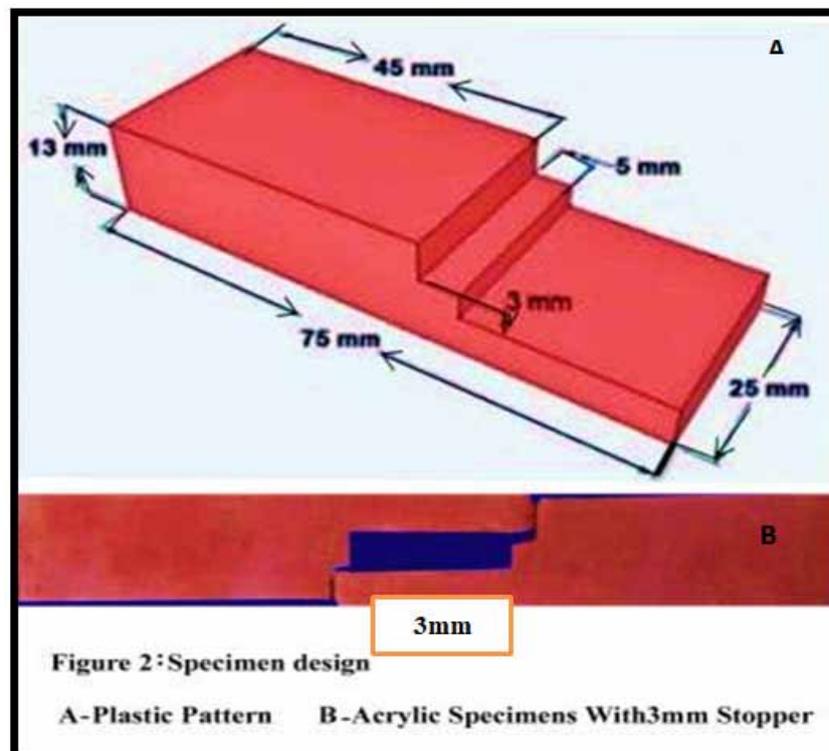
using universal Instron testing machine with suitable grips for the test specimens. The specimens subjected by using Instron testing machine at load cell capacity of (100 g) and crosshead speed equal to 0.5mm/min until failure occurred. The maximum load required for the failure was recorded. The shear bond strength value for each specimen was measured according to ASTM specification D-638m, 1986) using the formula below as in Figure (3-A).

$$\text{Bond strength (N/mm}^2\text{ )} = \frac{\text{Maximum load [force failure]}}{\text{Cross section area [surface area]}}$$

## Types of failure

The surfaces of bond failure between heat cure acrylic resin and acrylic soft liner were examined. (Salman, 2012) By means of an explorer naked eye for determining the type of failure (Alaa'a, 2013).

1. Adhesive (A): If the failure happened at the soft liner - denture base interface.
2. Cohesive (C): Soft liner material ruptured within itself.
3. Mixed (M): If part of the failure happened at the soft liner - denture base interface whereas the remaining part of the failure happened within the soft liner itself. (Khanna et al, 2015).



## Results

Table (1) shows that the highest mean value ( $0.693 \pm 0.068$ ) were obtained in group of 0.75 % Ag -Zn Zeolite, while the lowest mean value represented by control group ( $0.553 \pm 0.119$ ). The levene test demonstrated a non-significant difference ( $p > 0.05$ ) among studied groups, the one-way ANOVA result presented a highly significant difference ( $p < 0.01$ ) among all studies groups.

**Table 1: Descriptive statistic and testing homogeneity of variances (Levene test) and one-way ANOVA test.**

Groups	NO	Mean	Std. D	Std. E	Min	Maxi	Leven	ANOVA
control	10	0.553	0.119	0.038	0.396	0.755	0.119 Non sign	0.008 highly sign
Ag -Zn Zeolite 0.5 %	10	0.677	0.109	0.034	0.544	0.835		
Ag -Zn Zeolite 0.75 %	10	0.693	0.068	0.021	0.592	0.799		

2- The Least Significant Difference (LSD) test was accounted significant different in 0.5% Ag-Zn Zeolite group in comparison to with control group ( $p < 0.05$ ), as well as (Ag-Zn Zeolite 0.75%) group was accounted highly significant difference in comparison to control group ( $p < 0.01$ ), while comparison between (Ag-Zn Zeolite 0.5 % and Ag-Zn Zeolite 0.75 %) had recorded no significant different at  $p > 0.05$  as shown in table 2.

**Table 2: LSD test among groups of acrylic soft liner.**

(I) Group	(J) Group	Mean Diff. (I-J)	Sig.	C.S. (*)
Control	Ag -Zn Zeolite 0.5 %	-0.1235	0.011	S
	Ag -Zn Zeolite 0.75 %	-0.1397	0.005	HS
Ag -Zn Zeolite 0.5 %	Ag -Zn Zeolite 0.75 %	-0.0162	0.722	NS

### Mode of failure

The result of the mode of failure after shear bond testing shown the all groups of heat cure acrylic soft liner are an adhesive failure as in Figure (2-B).

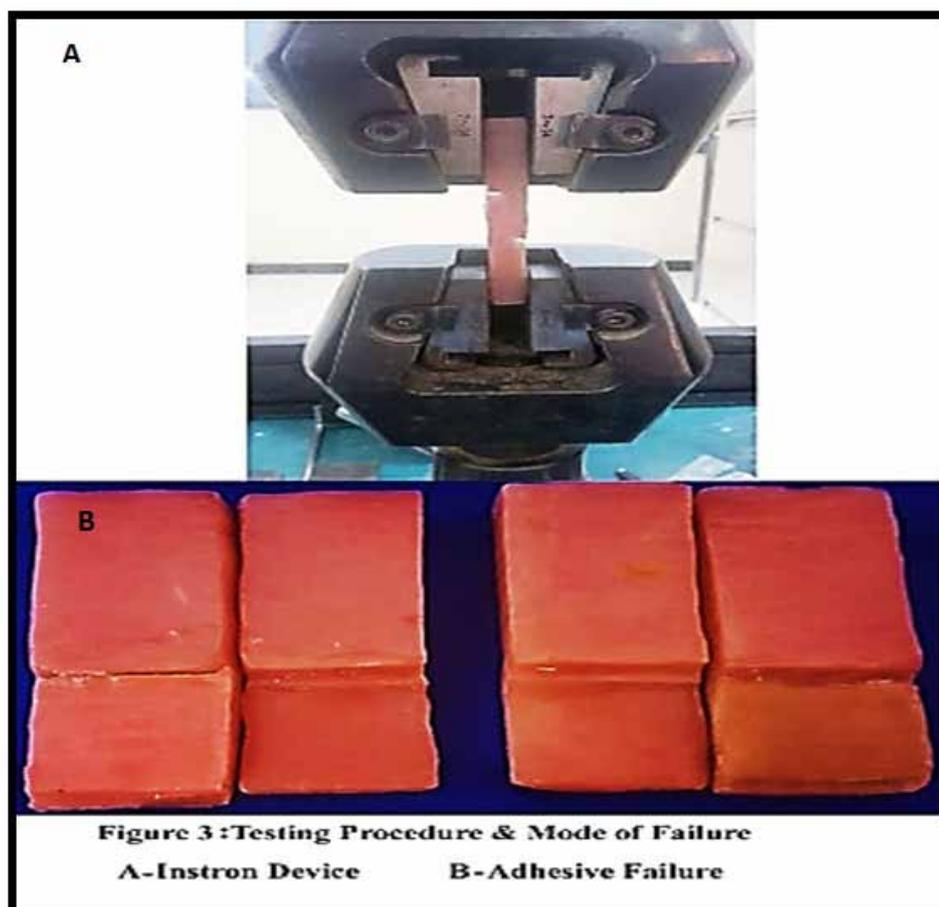
**Atomic Absorption Spectroscopy (AAS)** According to standard curve that represented the relation between standard concentrations for each  $Zn^{2+}$ ,  $Ag^{+}$  with the amount absorption and used special equation The amount of  $Zn^{2+}$  was 5.11 %, while the amount of  $Ag^{+}$  in Zeolite 13X was 0.44 %.

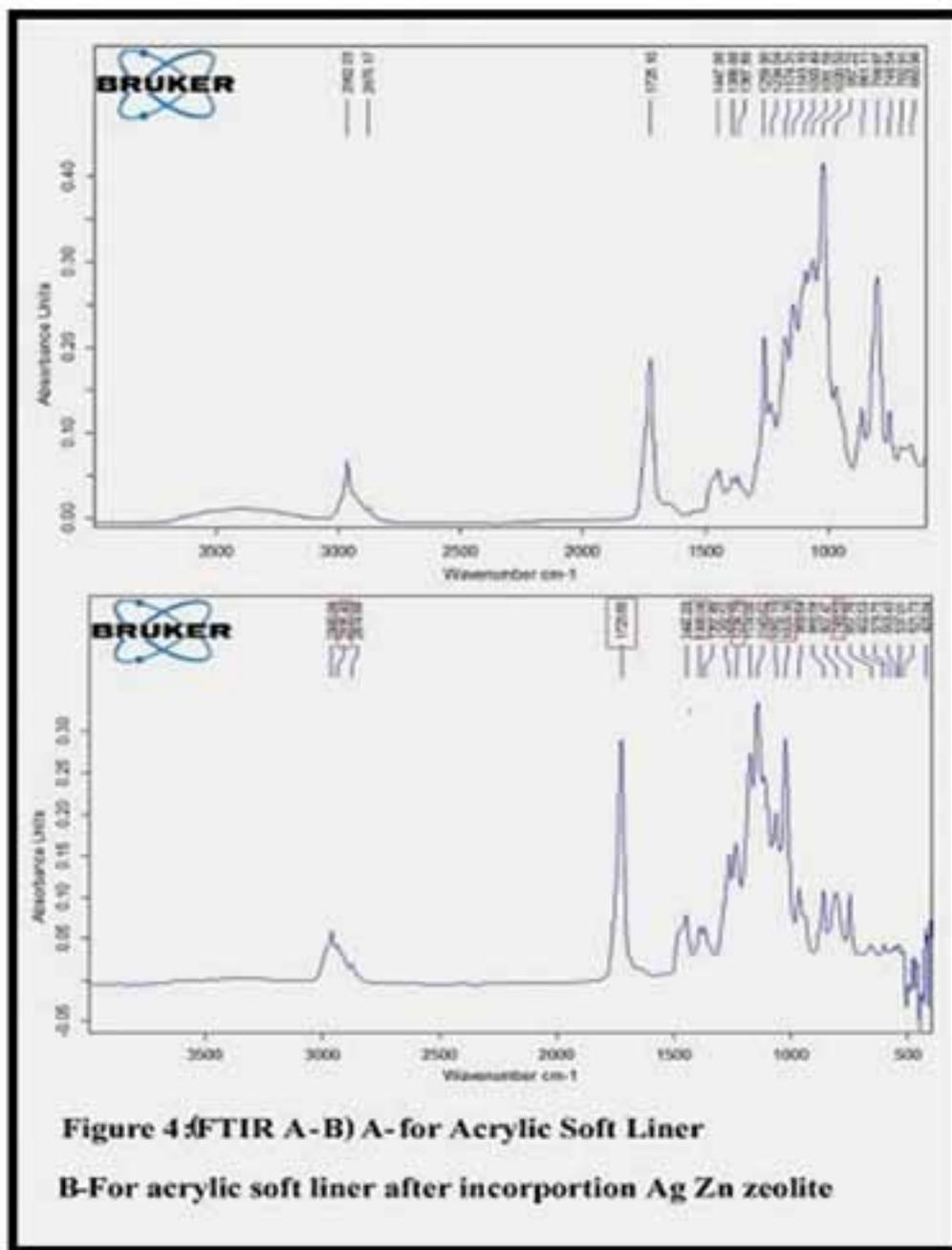
### X -RAY Fluorescence (XRF)

According to the result of XRF that determined the composition of pure zeolite 13X and after ion exchange with metal aqueous of  $Zn^{2+}$ ,  $Ag^{+}$  shown transfer the ion  $Zn^{2+}$ ,  $Ag^{+}$  within porous zeolite as shown in table 3.

**Table 3. (XRF) of Zeolite before and after ion exchange.**

Elements	Wt % before ion exchange	Wt % After ion exchange
O	46.30	45.22
Na	11.58	7.25
Mg	1.32	1.21
Al	14.51	13.42
Si	23.69	21.46
P	0.41	0.37
S	0.29	0.10
Cl	0.15	0
K	0.33	0.39
Ca	0.39	0.40
Ti	0.09	0.14
Fe	0.95	0.80
Ag	0	2.63
Zn	0	6.61
Total	100 %	100 %





### Discussion

This study involved the preparation and incorporation of Ag-Zn Zeolite into the acrylic soft liner that acts as antibacterial activity in this material (Singh et al., 2018). Anti-microbial zeolites have been utilized as filler with dental materials to stop or diminish bacterial, fungal and yeast contamination, at the same time, used to evaluate some mechanical properties (Casemiro et al, 2008).

## **Characterization of Ag- Zn Zeolite.**

### **Atomic Absorption Spectroscopy (AAS)**

The result of (AAS) Comparable ion concentration was gained for both silver and zinc ion for obtaining the same antimicrobial effectiveness of both ions, this agree with the work (Kaali et al, 2011), who investigated the applicability of different ion-exchanged zeolite as an antimicrobial additive in thermoplastic polyether- polyurethane.

### **Fourier transforms Infrared spectrophotometer (FTIR)**

The results of FTIR spectra for acrylic soft liner (ASL) materials before and after the incorporation of Ag-Zn zeolite recorded appear of new peaks and change intensity in some peaks when compared with FTIR spectrum of pure ASL. This may be attributed to the effect of Ag-Zn zeolite because of the interaction, distribution between the Ag-Zn zeolite and ASL, this could be explained the increase in shear bond strength. This coincidence with (Azzez, 2015) that incorporation the Ag-Zn Zeolite in to heat cure resins and revealed the effect it on some mechanical properties.

### **X ray fluorescence (XRF)**

The XRF used for determining the composition of Faujasite Zeolite 13 X and raw materials. After ion exchange, between Zeolite 13X and metals aqueous from (Zn<sup>2+</sup> and Ag<sup>+</sup>) acetate. The result shown Some of compositions of Zeolite 13X (Na, Mg, Al, Si, P, S, Fe) were decrease while increased in (K,Ca,Ti) and transmitted the (Ag<sup>+</sup>, Zn<sup>2+</sup>) in the composition of zeolite. Because of the zeolite has ability to ion exchange with metals aqueous from Zn<sup>2+</sup> and Ag<sup>+</sup> acetate and decrease the amount of concentration of some metals. It demonstrated that these metals replaced with (Zn<sup>2+</sup>, Ag<sup>+</sup>) ions. This result is agree with the work of (Aisiyah, et al, 2018) that indicated the (XRF) between non activation of natural zeolite and activation zeolite with HCL solution, which lead to decrease in some metals concentration by dissolved these metals in HCL solution.

### **Shear bond strength**

The shear bonding strength was used to evaluate the bonding strength between soft denture lining material and denture base because the forces that subjected into soft denture lining material in a clinical situation is represented more closely by tear and shear (Alaa, 2013). As in table (2), (3) The FTIR result appeared new peaks and change intensity for a soft liner with Ag-Zn zeolite due to interaction, uniform distribution between the acrylic soft liner and Ag-Zn Zeolite. Because of the ability of soft lining materials to flow highly which enhance the bond strength, allows the material to adapt easily to the bonding surface and offers good contact. Also the zeolite act as filler filling spaces between soft liner particles and increasing surface area for adhesion with denture base material. More ever ,the size sample (25mm length x 25 mm width) that used, is considered large and may be shared in the improvement bonding .This agrees with (Yasser & Fatah, 2017) Who evaluated the addition of zirconium Nanoparticles in the acrylic soft liner and found by addition zirconium Nanoparticles caused increase shear bond strength.

## Conclusions

1. Ion exchange between Ag-Zn Zeolite can be prepared by using aqueous solution of metal salts ion.
2. Atomic absorption spectroscopy, Fourier transform infrared spectroscopy, X-ray fluorescence, were found efficient methods for characterization of the synthesized material.
3. Fourier Transform infrared spectrophotometer was useful method to establish interaction between Ag-Zn zeolite and acrylic soft liner.
4. Incorporation of Ag-Zn Zeolite with 0.5% and 0.75% into heat soft liner had significant effect on shear bond strength.

## Conflict of interest

We are the authors (Huda Alaudeen Sadeq and Dr. Israa Mohammed Hummudi), state that the manuscript for this paper is original, and it has not been published previously (Part of Master Degree dissertation) and is not under consideration for publication elsewhere, and that the final version has been seen and approved by all authors.

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