

## Development of the properties of zinc polycarboxylate cement used as a basis for dental fillings using Alumina nanoparticles

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### ABSTRACT

Almost of dental materials do not appear a good seal from microorganisms. Thus, a microscopic space may exist at the interface between the packing material and the walls of the root end cavity, that is allowing microorganisms and their products to penetrate, and also to a perfect sealing capacity with biocompatibility, root fillers should ideally contain an antimicrobial efficacy. Therefore, this study targeted to estimate the antimicrobial activity of zinc oxide polycarboxylate cement (ZPCC) and its composites with aluminum oxide nanoparticles, Al<sub>2</sub>O<sub>3</sub>-ZPCC, & Green-Al<sub>2</sub>O<sub>3</sub>-ZPCC. The antimicrobial properties of ZPCC and its composites spread technology were tested against for *E. coli*, *S. aureus*, as well as *Candida albicans*. Based on the antimicrobial activity results, the addition of aluminium oxide nanoparticles to ZPCC improved its antimicrobial activity.

**Keywords:** Zinc oxide polycarboxylate cement ZPCC, Aluminum Oxide nanoparticles, Green chemistry, Al<sub>2</sub>O<sub>3</sub>-ZPCC, G-Al<sub>2</sub>O<sub>3</sub>-ZPCC, *E. coli*, *S. aureus*, *C. albicans*.

### INTRODUCTION

The commercially available dental cement materials vary in chemical constituent and that lead to possess them significantly various physical, biological and mechanical characteristics(1). The ZPCC are prepared by the heat-treated of zinc oxide and hydro poly acrylic acid (2). ZPCC is favored for luting fixed repairs in order to their good pulp compatibility. ZPCC did not caused primary ache that occurs after luting when casting repairs were introduced with Zinc oxide polycarboxylate cement ZPCC, because they did not cause an acute action. However, these cement materials contract at more area than another materials like zinc phosphate cements. This contraction, may be lead to cross-linking of poly acrylic acid chains with zinc atoms,

likely influences the safety of a bulk repairs(3,4). Recently, metal oxide nanobodies are have important uses in the several failed like ceramics, catalysis, semiconductors, medical science, space industry, batteries, capacitors, absorbents, agriculture, defense, textile, biological & chemical sensors, optoelectronics, as well as food industry [5-11]. Among all these, Al<sub>2</sub>O<sub>3</sub> NPs have drawn eminent attention in the advanced applications, like the designing and formulization of a new antimicrobial factors for biomedical implementations for the reason Al<sub>2</sub>O<sub>3</sub> NPs are more stable and bio-inert[12]. According to that, Al<sub>2</sub>O<sub>3</sub> NPs have been used in a number of branches, the important one is biomedical implementations in drug delivery, biosensors, and bio-filtration [13-15].

Al<sub>2</sub>O<sub>3</sub> NPs can be easily prepared by using various ways like hydrothermal [16], combustion [17], laser ablation [18], precipitation way [19], template way [20], mechanochemical [21], solvothermal [22], sol-gel [23], ball milling [24], microwave assisted [25], pechini method [26] and pyrolysis [27]. However, these prepare methods are very expensive, require long reaction time, and potentially hazardous. Therefore, these methods arise a bad effect on the ecosystem. This increases the pressing need to change or adjust chemical preparation approaches and development a sustainable, cost effective, non-toxic, clean, and environmental safety process throughout green approaches, by using capping agents such as plant extracts, fungi, algae, bacteria, biodegradable polymers and sugars, for the stabilization of Al<sub>2</sub>O<sub>3</sub> NPs. Alumina (Al<sub>2</sub>O<sub>3</sub>) nanoparticles has been used in different applications because of its various importance and beneficial properties. The chemical compound of alumina composed of aluminum and oxygen and most widely used ceramic materials among others ceramic material such as aluminum nitride, zirconia, silicon carbide, etc. Various applications that used alumina are as a biomedical implants, catalyst support and absorbents, fire retardants, polymer matrix composite, insulator and in clinical field, electronic fields, etc (28)

## EXPERIMENTAL PART

### Preparation of Al<sub>2</sub>O<sub>3</sub> nanoparticles

Al<sub>2</sub>O<sub>3</sub> nanoparticles were prepared according to a method used in the Mohamad et.al. Study (29).

### 2.2. Green Preparation of Al<sub>2</sub>O<sub>3</sub> nanoparticles

**The eucalyptus leaves extract was prepared** according to a method used in the Sarhan Study (30).

(0.3 g) of AlCl<sub>3</sub>.6H<sub>2</sub>O Al<sub>2</sub> (SO<sub>4</sub>)<sub>3</sub> .16H<sub>2</sub>O dissolved in 50 mL of DI and stirred quickly. (10 mL) of an extract put in a burette and gradually add to the mixture by dropping at about a room temperature. Then increase the temperature to about 80°C and gradually add (0.1M) of NaOH until the solution becomes basically (pH=8) and a precipitate is excite. Water and ethanol are used to wash the

sediment and remove pollutants. The precipitate is dried at 60°C for 3 hours, where nano powder of is formed, The formed powder (aluminum hydroxide) is calcination for 5 hours at 550°C, where nanopowder of aluminum oxide is prepared (30).

### 2.3- Preparation of Composites

To prepared Al<sub>2</sub>O<sub>3</sub>-ZPCC & G-Al<sub>2</sub>O<sub>3</sub>-ZPCC nanocomposites, (4g) of ZPCC mixing with (3 drops) of ZPCC liquid then added (1.4g) of Al<sub>2</sub>O<sub>3</sub> or G-Al<sub>2</sub>O<sub>3</sub> nanomaterials at a room temperature (31).

### 2.4- Antimicroorgansim influence

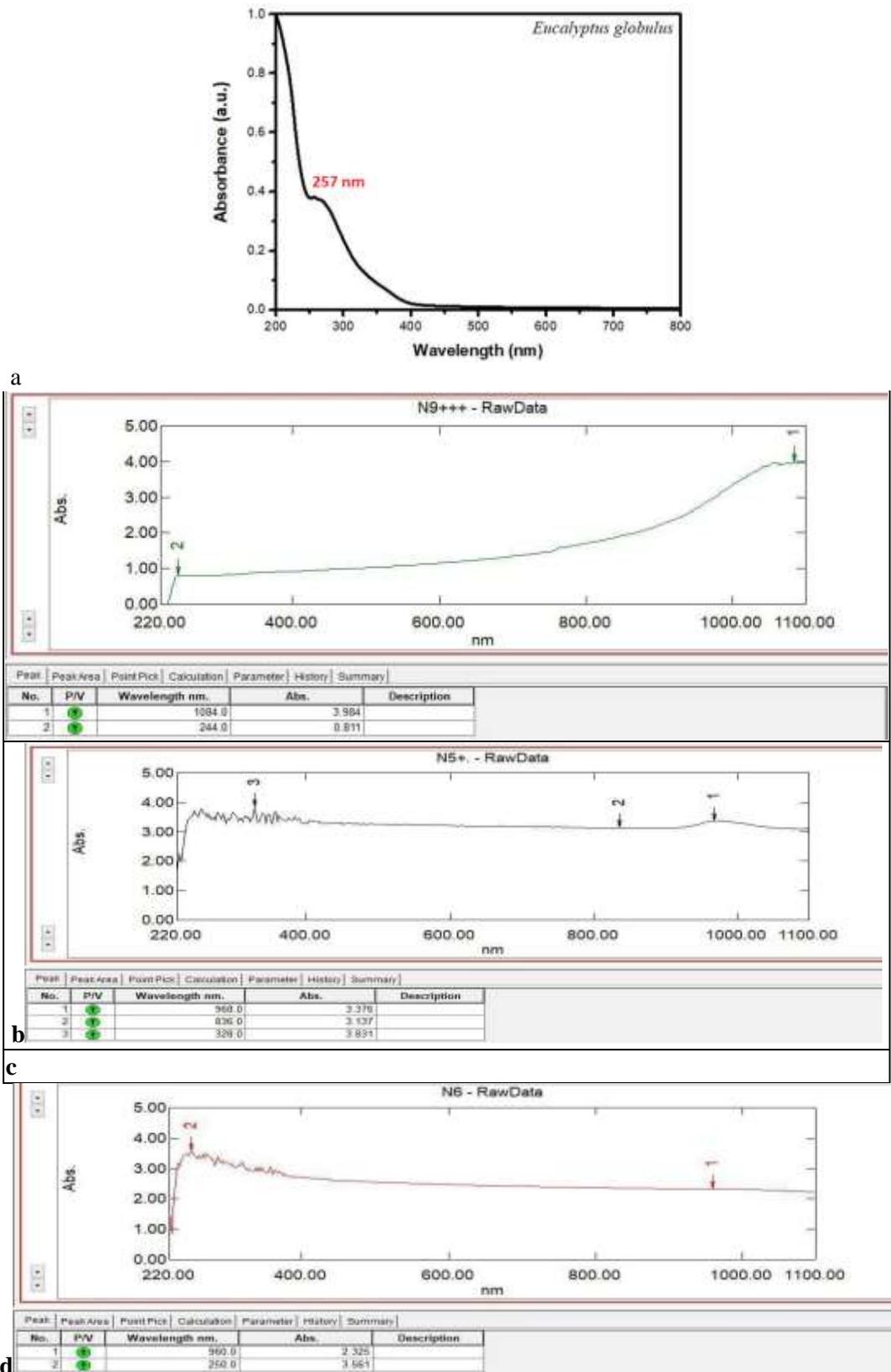
Antimicroorgansim influence of ZPCC, Al<sub>2</sub>O<sub>3</sub>-ZPCC & G-Al<sub>2</sub>O<sub>3</sub>-ZPCC nanocomposites were checked vs three various microorganisms, by according the way which described by (TM Media Titan Biotch Ltd) (32)

## RESULTS AND DISCUSSION

### 3.1- Characterization

Newly, in order to dominance the scale and form of nanomaterials, scientists by using individual phytochemicals for nanomaterials preparation, or known green chemistry. The presence of phytochemicals reduce the metal ions to nanometals. Therefore, the these compounds work, as a reducing and also as stabilizing factor. The UV-Vis spectroscopy displays the progress of the reaction. It showed an absorption peak associated with surface plasmon resonance, with electron vibrations collected in the conduction band by interaction with electromagnetic waves, which account for reduce the metal ions into nanometals. As it see in Fig. 1a, the spherical plant (eucalyptus) extract has one peak, which is 256nm. Flavonoids & tannins are the main phytochemical constituents of eucalyptus spherical extract that are bioactive agents and stabilizers, that cause the availability of hydroxide groups, for nanomaterial synthesis [33]. The phytochemical materials, being antioxidant as well as, free of hazardous chemicals are highly able of reducing & stabilizing of metal ion to the nano scale, and they can supply nanomaterials in various dimensional sizes and shapes [34]. An absorption maximum at 244 nm is shown in Fig. 1b, which presents the UV-Vis spectra of ZPCC.

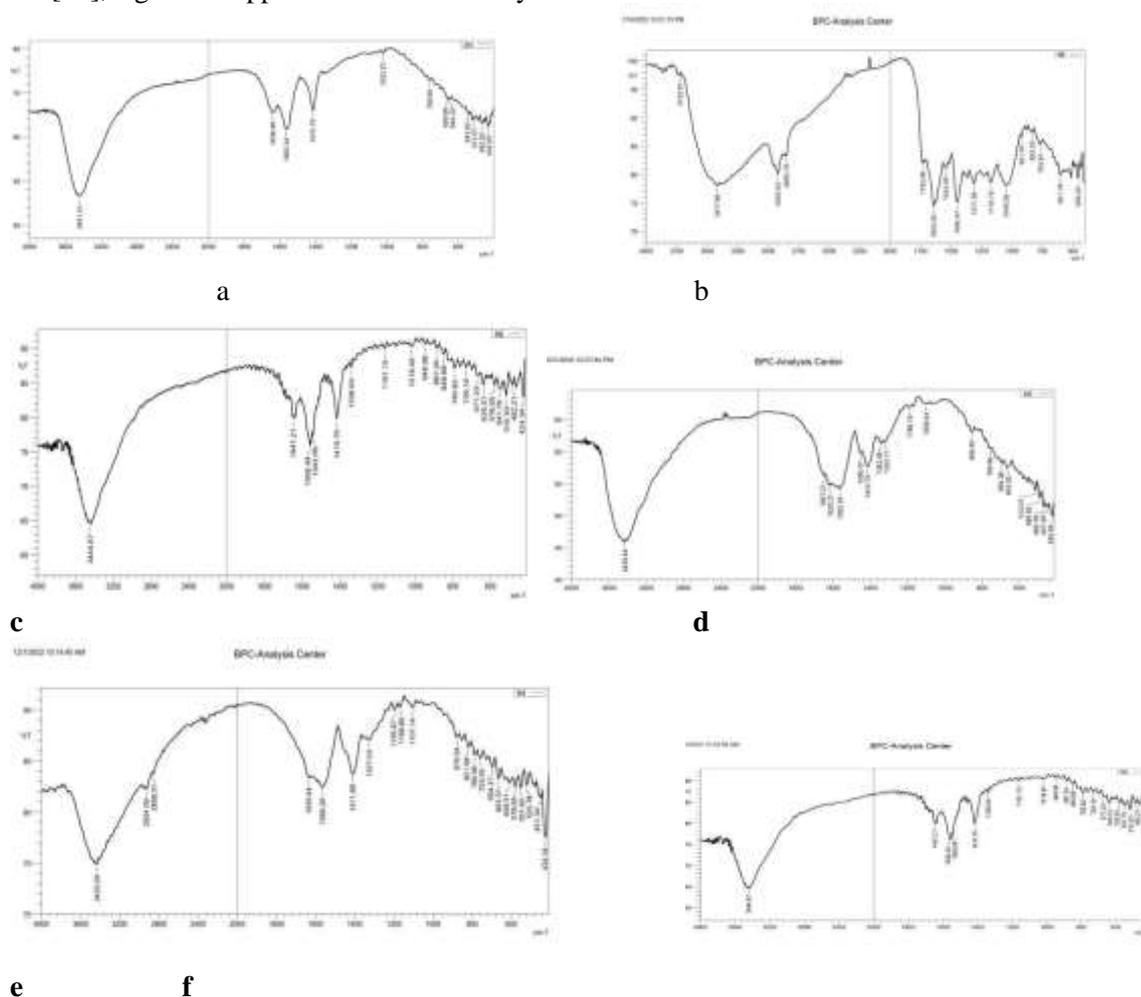
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**FIGURE 1:** UV-vis Pattern of a)Eucalyptus Leaves, b) ZPCC, c) Al<sub>2</sub>O<sub>3</sub>,d)Green-Al<sub>2</sub>O<sub>3</sub>

Eucalyptus leaf extract (Fig. 6a) contains many ligands. A peak at 3445 cm<sup>-1</sup> corresponds to vibration of hydroxyl group[35]. A peaks at 1570, 1417 and 1456 cm<sup>-1</sup> due to NH bonding of primary & secondary amides, stretching vibration of CN amides, or stretching CO of carboxylic acids, alcohols, ethers, as well as anhydrides [36]. The bands at 901 & 680 cm<sup>-1</sup> points to the exist of alkyl halide group. A broad band at 475 cm<sup>-1</sup> points to the exist of alcohol groups. Plant extract contains various phytochemicals[37], which may be responsible for the reduced the metal ions or the metal oxide and formed of nanomaterials [38]. Figure 2b appeared the FTIR analysis of (ZPCC), a peak at 1574.7 cm<sup>-1</sup>, is related to the stretching of absorption of CO of the COO group in a cements salt[39], figure 2c appeared the FTIR analysis of

Al<sub>2</sub>O<sub>3</sub>, it exhibits two broad peaks, the absorption peak at (507cm<sup>-1</sup>) is related to the octahedrals vibration (Al-O) bond (Al-O) and the peak at (626 cm<sup>-1</sup>) is related to the tetrahedral. The image approximately in the range 3150 cm<sup>-1</sup> shows on the near-image on the hydroxyl (OH) groups. A large band around 3400 cm<sup>-1</sup> is also observed, assigned to the -OH groups adsorbed on the nanoparticle surface [30]. After addition the alumina nanoparticles, the changes of absorption bands were seen easily Al<sub>2</sub>O<sub>3</sub>-ZPCC & G-Al<sub>2</sub>O<sub>3</sub>-ZPCC nanocomposites. Approximately at 1570 and 1420 cm<sup>-1</sup> peak intensity increased after nanomaterial amounts raised. The interaction between the cement and nanomaterials caused in the shifts of the cement absorption peaks.



**FIGURE 2:** FTIR Spectra Analysis of a)Eucalyptus Leaves, b)ZPCC, c)Al<sub>2</sub>O<sub>3</sub>-NPs, d)Green-Al<sub>2</sub>O<sub>3</sub>, e) Al<sub>2</sub>O<sub>3</sub>-ZPCC, & f) G-Al<sub>2</sub>O<sub>3</sub>-ZPCC.

### 3.2- Antimicrobial activity

An agar diffusion way used in here research because it is the most common mechanisms for evaluating the effect of antimicroorganisms. The pre-breeding period, which includes of keeping the inoculated culture environment at normal temperature for two hours, is an significant step in these way[40]. This way has several limitations because it can,t differentiate between bactericidal & bacteriostatic influences[41]. The efficacy and regions of inhibition are not correlated with the inhibitory influences of a substance only, but as well rely on the diffusibility of the objective through the environments[42]. Moreover, agents like incubation time, inoculum size, as well as good contact with the agar material may interfere with the effects too [40]. However, if almost of parameters are controlled properly, proportionate and reproducible results can be gained, and laterly the materials could be compared for their antimicroorganism influences under analogous experiances conditions[40, 41]. The selected examine microorganisms in our research were either real endodontic diseases or they related with treatment-resistant cases [40, .[43

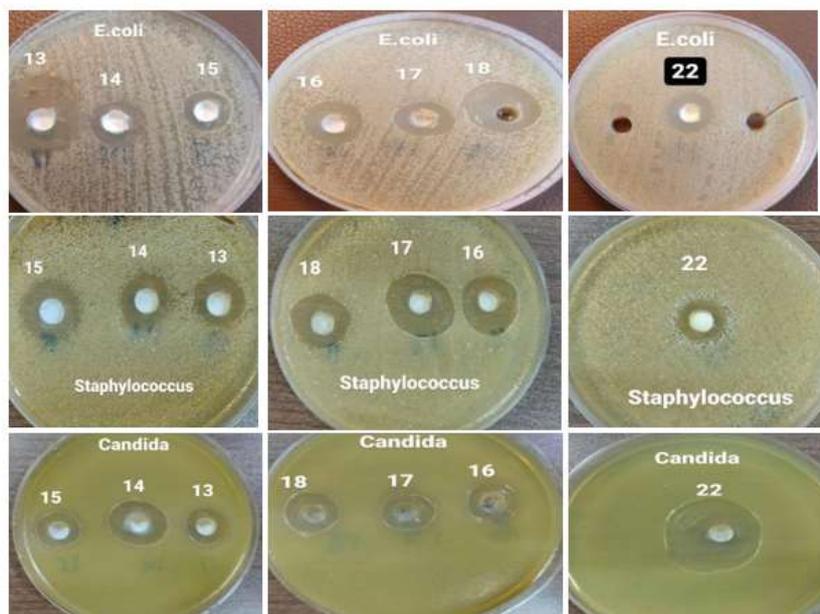
Although facultative & aerobic microbials are always secondary constituents of essential contagions, they have been found with higher frequency in states of prolonged treatment, in flare-ups, and in failed states [44]. E-coil, Staphylococcus aureus, and Candida albicans are potent microscopic organisms that may infect the root of canal [43-46]. The results of the

study(Table 1 and figure 3), showed influence of the antimicrobial of (ZPCC), Al<sub>2</sub>O<sub>3</sub>NPs-CEM, & green Al<sub>2</sub>O<sub>3</sub>NPs-CEM exhibited that the addition of different amounts of nanoparticles to ZPCC improved its antimicrobial effecting versus E-coil, S. aureus, & C. albicans. However, the antimicrobial efficacy of S. aureus did not varying. The enhancement of antimicroorganisms efficiency was significantly more for E. coli but lower against S. aureus and C. albicans, which may lead to a few .clinical improvement

The antimicroorganisms efficiency of ZPCC appears to be related with raise in pH data. The pH of ZPCC about ten, and increasing to about with addition nanoparticles[47]. It is 12.0 recognised that pH scales up to about 12 can stop the growth of almost microbials[48]. The antifungal activity of ZPCC can be imputed to its more pH or to the substances releasing from the ZPCC [49]. Small scale of nanoparticles may inhibit the growing of microbials higher than ions at the similar concentrations of the material Particle size was also associated with .[51 ,50] antimicrobial effect, a small bodies appeared high bactericidal influence than large bodies Figure 4, shows comparison of .[52,53] antimicroorganisms efficiency in terms of increasing in the zone inhibitors between ZPCC Al<sub>2</sub>O<sub>3</sub>-ZPCC nanocomposites , while figure & shows comparison of antimicroorganisms ,5 efficiency in terms of increasing in the zone inhibitors between ZPCC & G-Al<sub>2</sub>O<sub>3</sub>-ZPCC, .composites

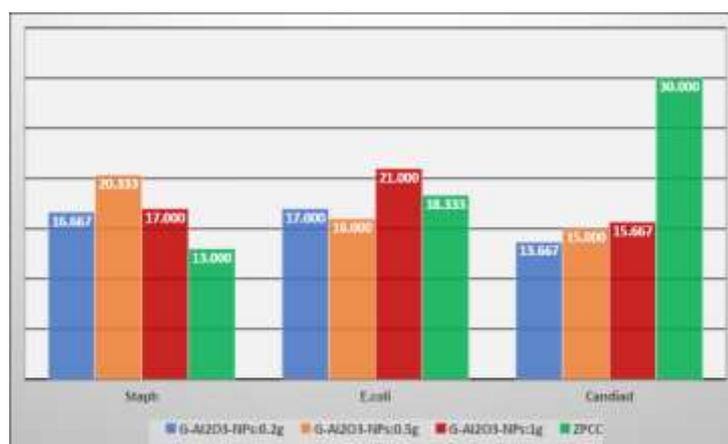
**TABLE 1:** Antimicroorganisms efficiency in terms of increasing in the zone inhibitors between ZPCC, Al<sub>2</sub>O<sub>3</sub>-ZPCC & G-Al<sub>2</sub>O<sub>3</sub>-ZPCC.

Microbiall species	ZPCC	Al <sub>2</sub> O <sub>3</sub> -ZPCC 0.2g	Al <sub>2</sub> O <sub>3</sub> -ZPCC 0.5g	Al <sub>2</sub> O <sub>3</sub> -ZPCC 1g	G-Al <sub>2</sub> O <sub>3</sub> -ZPCC 0.2g	G-Al <sub>2</sub> O <sub>3</sub> -ZPCC 0.5g	G-Al <sub>2</sub> O <sub>3</sub> -ZPCC 1g
E-coli	13	19	18	19	16	21	16
S.aureus	19	17	13	16	20	17	17
C.albicans	30	14	14	16	15	16	17



**FIGURE 3:** Antimicroorganisms efficiency in terms of increasing in the zone inhibitors between ZPCC , Al<sub>2</sub>O<sub>3</sub>- ZPCC, & G- Al<sub>2</sub>O<sub>3</sub>- ZPCC composites.

**FIGURE 4.** Comparison of antimicroorganisms efficiency in terms of increasing in the zone inhibitors between ZPCC & Al<sub>2</sub>O<sub>3</sub>-ZPCC composites.



**FIGURE 5.** Comparison of antimicroorganisms efficiency in terms of increasing in the zone inhibitors between ZPCC , & G-Al<sub>2</sub>O<sub>3</sub>NPs- ZPCC composites.

### Statistical analysis

Data analysis methods utilized the statistical software package for the social sciences (SPSS) version seventeen (Chicago, USA). The experience for normality used the Shapiro-Wilk test, while the test for homogeneity utilized the Levene exam. All data were showed as mean ± SD. The means & standard deviations of growth inhibition diameters versus various examined microbials are shown in a figure 5 and table

We use the independent t-test to check for .2 significant differences between groups or not using 3 different measures (0.2, 0.5 and 1g). There are significant differences between NPs and ZPCC in Candida with 0.2, 0.5 and 1 g, and in Escherichia coli with 1 g, and the statistical analysis showed normal distribution of growth inhibition diameters in ZPCC, Al<sub>2</sub>O<sub>3</sub>-ZPCC, & G-Al<sub>2</sub>O<sub>3</sub>-ZPCC composites groups versus .examined microorganisms

**TABLE 2.** Mean & standard deviation of growth inhibition diameters versus examined microorganisms per mm of ZPCC & Al<sub>2</sub>O<sub>3</sub>-ZPCC composite.

Bacterial	0.2g			0.5g			1g		
	E.coli	Staph	Candiad	E.coli	Staph	Candiad	E.coli	Staph	Candiad
Al <sub>2</sub> O <sub>3</sub> -NPs	15.667	18.667	13.667	12.333	17.667	20.667	19.000	15.333	15.000
ZPCC	13.000	18.333	30.000	13.000	18.333	30.000	13.000	18.333	30.000
p-value	0.05598	0.75516	0.00008**	0.54128	0.54185	0.00073**	0.00388**	0.03994*	0.00012**

\* The difference is considered to be statistically significant under 0.05.

\*\* The difference is considered to be highly statistically significant under 0.01.

Figure 5. Comparison of mean values of growth inhibition diameters versus examined microorganisms per millimeter of ZPCC & Al<sub>2</sub>O<sub>3</sub>-ZPCC composite.

At the same way, the means and standard deviations of growth inhibition diameters versus various examined microbials are shown in Table 3 and figure 6. We use the independent t-test to check for significant differences between groups

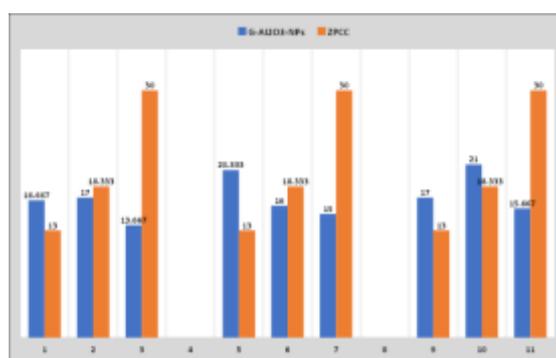
or not using 3 different measures (0.2g, 0.5g and 1g). There are significant differences between G-Al<sub>2</sub>O<sub>3</sub>-NPs and ZPCC in E. coli with 0.2 g, 0.5 and 1 g, and in Staph. with 1.0 g, and the statistical analysis showed normal distribution of growth inhibition diameters in ZPCC and Al<sub>2</sub>O<sub>3</sub>NPs- ZPCC, composites groups versus examined microorganisms.

**TABLE 3.** Mean & standard deviation of growth inhibition diameters versus examined microorganisms per mm of ZPCC & G-Al<sub>2</sub>O<sub>3</sub>-ZPCC composite.

Bacterial	0.2g			0.5g			1g		
	E.coli	Staph	Candiad	E.coli	Staph	Candiad	E.coli	Staph	Candiad
G-Al <sub>2</sub> O <sub>3</sub> -NPs	16.667	17.000	13.667	20.333	16.000	15.000	17.000	21.000	15.667
ZPCC	13.000	18.333	30.000	13.000	18.333	30.000	13.000	18.333	30.000
p-value	0.02145*	0.25339	0.00008**	0.00184**	0.07999	0.00012**	0.01613*	0.05598	0.00014**

\* The difference is considered to be statistically significant under 0.05.

\*\* The difference is considered to be highly statistically significant under 0.01.



**FIGURE 6.** Comparison of mean values of growth inhibition diameters versus examined microorganisms per mm of ZPCC & G-Al<sub>2</sub>O<sub>3</sub>-ZPCC composite

## CONCLUSION

In this study, data collected from agar diffusion assays, display the addition of several quantities of aluminum oxide NPs (which prepared by using chemical and green methods) to ZPCC to give Al<sub>2</sub>O<sub>3</sub>-ZPCC, and G-Al<sub>2</sub>O<sub>3</sub>-ZPCC composites., can be a valuable alternative to increasing in the antimicroorganism activities of this substance against a clinical isolate of 3 important microorganisms utilized in our research, E-coil, S.aureus, & C. Albicans. on the antimicroorganism efficiency of endodontic cement. Based on the our results, the addition of Aluminum oxide nanoparticles to ZPCC improved its antimicrobial activity.

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