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Impact of different concentration of CaF_2 on some properties of acrylic resin material and electrochemical behavior in saliva

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Aim: of the study was to study the consequences of different ratio of calcium fluoride on some properties of heat acrylic resin material (transverse strength and surface roughness)in addition study its electrochemical behavior on the saliva and blood.

Material and Methods: 40 samples have been intended according to ADA specification dimensions (65x10x2.5)mm length, width and thickness respectively were prepared from acrylic resin material,10 samples without additive (Control) and 30 samples incorporated with CaF_2 in different concentrations (1%, 2%and 3%), Transverse strength tested by Instron Universal machine , Roughness test was done by TR 220 portable tester and electrochemical behavior by using cyclic voltammetry.

Results: control group recorded the minimum mean value for transverse strength and roughness test ,while 3%CaF_2 recorded the highest value , for all test the significant were differences between control and CaF_2 groups .electrochemical behavior study showed in the acidic pH medium of artificial saliva had oxidative behavior and in alkaline pH medium of artificial saliva had antioxidant property

Conclusion: The addition of CaF_2 showed significant improved in transverse strength ,but had adverse effect on roughness in comparison to control group, calcium fluoride be reliable compound with both saliva and blood and safe for incorporation with dental prostheses at (1%,2%and3%).

Keywords: CaF2 , acrylic resin , electrochemical, properties

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Introduction

The non-metallic materials using in manufacturing of prosthesis such as space maintainer and denture have a long tradition in dentistry. [1,2] However, studies and a notable challenge demonstrated to advance their properties and susceptibility to fungal growth, compromising their integrity by incorporating various additives that will enhance the properties of acrylic material. Delves into the innovative integration of antibacterial material into acrylic resin, focusing on the consequential evaluation of resin properties. Discern the effects of this incorporation, providing valuable insights for applications that demand acrylic resin materials with heightened on the advancement of its properties . Patients who wear appliance of acrylic resin material especially space maintainer have to control oral hygiene especially for which the causative factor for decay of tooth was the bacteria. [3] Many fluoride salts used in hygiene products. [4,5] that release fluoride ion into the mouth enhancing tooth strength through deposition of fluoride ion on the tooth surface due to increasing fluoride ion in saliva. [6,7]

Calcium fluoride is the first source of fluorine in world , it is in organic compound of element calcium and fluorine. [8] Incorporation of fluoride into acrylic material assimilate as fluoride release device that was benefits for pediatric wearing space maintainer, there were some studies tested the properties of acrylic after incorporated with different fluoride salt. [9-13] and some studied assess the percentage of fluoride release after incorporation with acrylic resin materials. [14-17] To the best of knowledge there was no enough information regarding the effect of CaF_2 on the acrylic resin properties utilized for space maintainer and denture, although this material had been found to showed protract and the longest time duration for releasing fluoride ion up to 6 months from acrylic plates that incorporated with different concentration of CaF_2 in comparison to other fluoride compounds. [16,17]

The transverse strength test was valuable in comparing materials of space maintainer and denture in which the type of stress applied to the prosthesis through mastication.[18] Surface hardness of material most important properties to assume the dense material that more resistance to wear and degradation of the surface.[19] and its related to surface roughness which has an effect on the performance of a product as well as its quality specifically to wear and fatigue resistance. [20-22] Since fluoride was used as anticaries and antibacterial agent, there was still a lack in knowledge that concerning the incorporation of calcium fluoride into acrylic material so the aim of study specifically to assess the transverse strength and surface roughness of acrylic resin space maintainer and denture after incorporating with divergent concentration of CaF_2 focusing on determining the optimal CaF_2 percentage without compromising these properties, hypothesizing an improvement in properties. Despite potential benefits, there was a gap in research regarding this impact on properties of resin material in addition to study the nature of calcium fluoride its interaction with saliva and blood, in order to understand its behavior and effectiveness in these biological environments

Materials and Methods

Preparation of test specimens

The designing of samples were done according to specification for test and materials. Metal patterns design by (CNC) for study test were prepared to obtain samples from heat cure acrylic resin. The design of transverse strength and surface roughness were same which was rectangular shaped according to (ADA No.12, 1999). [23] the dimensions was (65x10x2.5 mm) length, width and thickness subsequently.

Specimens grouping

40 samples were prepared for this study constructed from control group without adding CaF_2 (0%), The experiment samples were divided into three groups based on CaF_2 concentration 1%, 2% and 3% each group (10) specimens , these concentrations was add to monomer of acrylic, these percentages were deducted from the volume of the powder of polymer to obtain accurate P/L ratio of 1%, 2% and 3% calcium fluoride.

Method of incorporation of CaF2 into acrylic resin material

In this study Powder and liquid of heat cured acrylic resin used was Vertex Netherland. Mixing together proportional to the instruction of manufacturer's using a container with spatula , the mixing procedure done in clean glass jar for control group weighting 15 g of acrylic powder and mixed with 10 ml of monomer liquid according to manufactural information, and for experimental specimens used percentage of the CaF_2 powder(1%,2%,3%)CaF_2 had been incorporated into monomer of acrylic, these percentages were deducted from the volume of the powder of polymer to obtain accurate P/L ratio, the CaF_2 was mixed with liquid (monomer) of acrylic resin about 20 seconds. [24] by used sonication apparatus probe at 60 KHz and 120 W to ensure homogeneity done in complete manner, then adding the powder (polymer) , the mixture mixed then put the mixture in the container and left till reached the dough stage.

Specimen preparation:

The molds prepared from using the standard flasking technique. [23]Packing, curing procedure, finishing and polishing were done according to conventional methods. [25]

transverse Strength Test

The transverse strength of a materials was obtained by one loads a simple beam that supported at each end with applying a load in middle and the test named three-point bending procedure (3PB). With a stainless-steel rod in the shape of a chisel and a crosshead at speed of 0.5 mm/min, the samples loaded till fracture and the fracture load was recorded by an Instron machine (mode lwdw50) for the shear bond strength (ISO TR 11405). The load cell sated at 100 kg, and the force at fracture (F) and the sticky surface area (S) were computed and converted to Mpa. [26] as in the following.

B.S = F / …… (Equation2)

B.S = Bond strength (N/mm^2) or (MPa)

F=force at failure $S= (\pi / 4) \times D^2$; π =22/7 or 3.14

D (diameter) = 5mm, S = 19.64 mm^2.

Surface roughness test

Rectangular shaped specimen printed the dimensions was(65x10x2.5 mm) length, width and thickness subsequently by printer , Surface roughness test was performed using surface roughness profilometer tester (TR200) with 0.001 micrometer accuracy at (University of Technology, Materials Engineering Department), This tester contained a diamond sensitive needle (stylus) using to track the irregularities on the surface. Three separated locations on the specimen's surface were just touched by the stylus to have three readings for each sample, so according to profilometer instructions; the sample was located on a stable, rigid surface and the stylus should be allowed to contact the first point, then it was moved for 11 mm across the sample, the readings appeared on the digital scale in a spontaneous manner. Later, a roughness values were determined by calculating the mean values of these reading in µm .[27,28]

Cyclic voltammetry

Preparing of artificial saliva was done by scientific lab Shafeeq comp (Iraq), 0.1M from HCl and 0.1M from NaOH used as a buffering solution . The cyclic voltammetry cell technique was prone by add 10ml from artificial saliva in the cell then immersed the working electrode(the glassy carbon electrode),by used Ag/AgCl the reference electrode and used the platinum wire as counter electrode, the 3 electrodes linked to a potentiated (potentiated/glvanostat) by NuVant System EZstat (U.S.A.), and a cyclic voltammogram calculated the results CNT (Fluka, 98 %), human healthy blood samples receiving from Iraqi Blood Bank in Baghdad Medicine City. Deionized water used for the preparation of aqueous solutions.. Otherwise solvents and chemicals of annular grade used as receiving from the manufacturer . For the preparation of aqueous solutions , deionized water used. All solutions deaeration with oxygen free nitrogen gas about 10–15 min before measurement. [29,30]

Results

Transverse strength

The descriptive statistic as showed in Table 1, showed the transverse strength of resin increased significantly with addition of CaF_2. the control group recorded the minimum strength value. The average strength increased even further in groups IV (3% CaF2)

Table 1:Statistical description and ANOVA analysis of the study sample for the surface transverse sample

Groups	N	Mean	Std. Deviation	Std. Error	Minimum	Maximum	Anova P-value
Control CaF_2 0%	10	89.4	2.31	.73333	86.00	92.00	0.013 S
1%CaF_2	10	91	1.69	.53748	88.00	93.00	
2%CaF_2	10	91.6	1.71	.54160	88.00	93.00	
3%CaF_2	10	92	1.24	.39441	89.00	93.00	

S;significant.

Table(2) showed post hoc analysis for action of CaF_2 1%, 2% and 3% concentration on transverse strength test of resin materials, the differences were significant between control group with all groups of CaF2 but there was no significant between CaF2 groups

Table 2: Multiple Comparisons analysis of the study sample for the surface transverse sample

Groups	Surfacetest	Mean Difference	Std. Error	Sig.	95% Confidence Interval	
					Lower Bound	Upper Bound
Control CaF_2 0%	1%CaF_2	-1.60000	.79861	.053	-3.2197	.0197
	2%CaF_2	-2.20000*	.79861	.009	-3.8197	-.5803
	3%CaF_2	-2.60000*	.79861	.002	-4.2197	-.9803
1%CaF_2	2%CaF_2	-.60000	.79861	.457	-2.2197	1.0197
	3%CaF_2	-1.00000	.79861	.219	-2.6197	.6197
2%CaF_2	3%CaF_2	-.40000	.79861	.620	-2.0197	1.2197

*. The mean difference is significant at the 0.05 level.

Surface roughness Test

Table (3) showed the descriptive analysis of all groups Control group showed the minimum mean value for roughness test while the group 3%CaF_2 recorded the maximum mean value for roughness test. ANOVA test showed the differences was significant among groups p-value ≤ 0.0001.

Table 3: Statistical description of the study sample for the surface roughness sample

Groups	N	Mean	SD	SE	Min.	Max.	Anova P-value
Control CaF2 0%	10	0.12	0.07	.02327	.03	.27	0.0001≤ H.S
1%CaF_2	10	0.33	0.05	.01695	.23	.39	
2%CaF_2	10	0.39	0.02	.00947	.36	.46	
3%CaF_2	10	0.44	0.04	.01413	.40	.51	

Table (4) showed post hoc analysis for the action of CaF_2 1%, 2% and 3% concentration on roughness of resin materials, the differences were significant between all groups .

Table 4: Multiple Comparisons analysis of the study sample for the surface roughness sample

Groups	groups	Mean Difference	Std. Error	Sig.	95% Confidence Interval	
					Lower Bound	Upper Bound
Control 0% CaF_2	1%CaF_2	-.20810*	.02364	.000	-.2561	-.1601
	2%CaF_2	-.26360*	.02364	.000	-.3116	-.2156
	3%CaF_2	-.31660*	.02364	.000	-.3646	-.2686
1%CaF_2	2%CaF_2	-.05550*	.02364	.025	-.1035	-.0075
	3%CaF_2	-.10850*	.02364	.000	-.1565	-.0605
2%CaF_2	3%CaF_2	-.05300*	.02364	.031	-.1010	-.0050

*. The mean difference is significant at the 0.05 level.

Electrochemical properties

The electrochemical properties of the different concentrations in aqueous solutions of CaF_2 , the voltammogram of CaF_2 in artificial saliva, has (oxidation peak current at a potential of +1.25 V) and enhanced the oxidation peak at +1.5 V. The CaF_2 of the oxidation process in the artificial saliva acts as electro-catalyst.

Effect of divergent pH of artificial saliva and blood

The effect of divergent pH of artificial saliva was studied on the oxidation peak current of CaF_2. When calcium fluoride (CaF_2) is introduced into saliva, a weak oxidation reaction occurs, gradually releasing calcium ions (Ca^{2+}) and fluoride ions (F^-) ,benefits of the reaction in enhancing dental health , the oxidation peaks of the CaF_2 appeared at the acidic pH (3-6) ,while disappearance at the alkaline pH (7-9).

In this study, the effect of CaF_2 in medium of blood studied by using cyclic voltametric technique. Figure 1 showed high current peak of CaF_2 ions in blood medium CaF_2 acts as an antioxidant in an alkaline environment (like blood). In the alkaline medium (blood), a weak reaction occurs without side effects, indicating safety for medical applications such as dental prostheses (space maintainer and denture) or restorative materials. In certain conditions, Fig 2, such as in smokers or poisoning cases, the blood pH may become more acidic, Therefore, the blood pH was gradually altered during the test to study the reaction of CaF_2 under these conditions. Calcium fluoride is biocompatible with both saliva and blood.

Discussion

Most commonly used for space maintainer and denture was the heat cure acrylic resins. The essential limitation was act as microorganism's reservoir. The adherence of microorganisms could be minimized using chemical modification of the surface charge of denture base resin. [31]Several attempts had been conducted to reduce this ability by many additives that help in resolving this problem.[32,33] In this study using addition of different concentration of calcium fluoride (1,2, and,3)% to assess the properties of acrylic material using especially in construction of space maintainer , after incorporation with these concentration . CaF_2 is A white solid naturally crystalline compound, its molecular weight is 78.07, its density is 3.18 g/cm3. Calcium fluoride is also considered a salt and has an important role in eliminating harmful bacteria in the mouth. [34] The concentration of fluoride salt that mixed with monomer of heat cured acrylic should considered the level of fluoride release and consider the less effect of fluoride on the acrylic properties , the greater fluoride concentration acrylic samples was the great fluoride release. [35] However more studies need to conduct influence of increasing fluoride concentration on the properties of space maintainer and denture.

Transverse strength refers to the ability of a material withstanding the forces acting perpendicular to its longitudinal axis. Modifying the acrylic resin prosthesis was one method of strengthening acrylic material. Factors that can affect transverse strength include the material's composition, structure, and processing method.[36]

Roughness test one of the important method useful to determine the mechanical properties of acrylic resin materials, also its important test in prevent the microorganism to attached to denture base.[37,38,39] In this study showed that the surface roughness increase when concentration of CaF_2 increased this was might be that the fluoride salt interfering within the polymerization. This happens via the exposure of polymer beads which result to an increased in the porosity and increasing in roughness. [40] The Fluoride addition to the acrylic resin result in the intermolecular interaction also the fluoride when presence in methacrylic polymers results on divergent intermolecular distances that affect the roughness of this material .The fluoride incorporation into resins material result in incompatibility by a large divergent in polarity between the low polarity of the dental resin and the ionic fluoride .This incompatibility causing phase separation with the resin so fluoride releases within time. This decrease in cohesive energy which minimized the effect of polymer chain entanglement. [41,42] The result was in agreement with study by (Srithongsuk et al., 2012)[17] , (Rashid, 2015) [10] and (Jitaluk et al., 2022)[43],which showed that acrylic was seen to be porous after the addition of different fluoride salts ,increase porosity of acrylic result in the majority of acrylic resin materials hardness reduces, but disagree with(Ali,2014)[9] who was reported increased in hardness after fluoride salts incorporation to acrylic resin, this vice versa in results could be due to differences in fluoride addition procedures as well as the type of fluoride salts was differ, CaF_2 could be help to make resins less hard, which might be beneficial in applications that require fracture resistance such as soft lining materials.

Using effect of divergent pH of the medium of artificial saliva was studied on the oxidation peak current of CaF_2. And showed oxidation peaks of the CaF_2 was appeare in the acidic pH (3-6) ,while disappearance in the alkaline pH (7.9). Artificial Saliva (slightly acidic medium pH around 6.5-7.5) when calcium fluoride (CaF_2) was introduced into saliva, a weak oxidation reaction occurs, gradually releasing calcium ions (Ca^{2+}) and fluoride ions(F^-). This reaction had benefit in enhancing dental health by fluoride ions that help in the remineralization of tooth enamel, making it more resistant to decay and useful in construction of space maintainer. [44]

Blood (alkaline medium pH around 7.3,7.4) when calcium fluoride (CaF_2) was introduced into blood, a weak reaction occurs without noticeable side effects indicating biocompatibility. In certain conditions, such as in smokers or poisoning cases, the blood pH may become more acidic. Therefore, the blood pH was gradually altered during the test to study the reaction of CaF_2 under these conditions. Calcium fluoride is biocompatible with both saliva and blood, making it suitable for medical applications such as dental prostheses (space maintainer and denture) or restorative materials regarding percentages related to this study .[44]

Conclusion

The addition of CaF_2 showed significant improved in transverse strength of acrylic prosthesis (space maintainer and denture), but had adverse effect on roughness in comparison to control group calcium fluoride be reliable compound with both saliva and blood and safe for incorporation with dental prostheses especially space maintainer regarding percentages related to this study.

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