Effect of Incorporation of Calcium And Fluoride Salts on Transverse Strength of Acrylic Resin

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Aims: The aims of this investigation were to determine the effect of incorporation of CaCO$_3$ from egg shell and Fluoride salt NaF in different concentrations on transverse strength of RESPAL® NF(VEIN 24) heat cured acrylic resin denture base.  

Materials and Methods: 21 samples of heat cured acrylic resin were prepared, divided it's into 7 groups; the 1st group Control HCAR , the 2nd group HCAR mixed with 2.5% CaCO$_3$, the 3rd group HCAR mixed with 5% CaCO$_3$, the 4th group HCAR mixed with 10% CaCO$_3$ and the 5th group HCAR mixed with 2.5% NaF, the 6th group HCAR mixed with 5% NaF, the 7th group HCAR mixed with 10% NaF . The samples were tested after 48 hours from preparation for transverse strength test. 

Results: One way ANOVA and Duncan Multiple Analysis rang test were used and showed significant differences in the transverse strength between the two groups (CaCO$_3$ and NaF), and significant differences between control and CaCO$_3$ while no significance between control and NaF groups. 

Conclusions: It was concluded that adding NaF to HCAR led to an increase in strength of denture base and significant decrease in strength when adding CaCO$_3$ to HCAR .This was within acceptable range of American Dental Association specification no.12 ,1975.

Key words: Acrylic resin ,Transverse strength , NaF , CaCO$_3$.

INTRODUCTION

Acrylic resin is the most employed material in the construction of removable complete denture, used since 1930. Characterized by being strong, having a satisfactory optical property coping oral tissue appearance, showing low water sorption and solubility and having a good dimensional stability.$^{(1,2)}$

Calcium carbonate, a pharmaceutical excipient, is widely used as diluent in solid dosage forms. Also used as a base for medicinal and dental preparations, a buffering and dissolution aid for dispersible tablets, a food additive and a calcium supplement.$^{(3)}$

Thapen and Bourgeois, 1994 found that the shell is approximately 11% of the total weight of the egg and it presents contents of: calcium carbonate (94%), calcium...
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phosphate (1%), magnesium carbonate (1%) and organic substances (4%). So, egg shell is a rich source of mineral salts, mainly calcium carbonate.\(^4\)

Fluoride salts are used to enhance the strength of teeth by the formation of fluoroapatite, a naturally occurring component of tooth enamel.\(^5,6\)

Administration of sodium fluoride and calcium carbonate produce an increase in bone mass and is a useful adjunct treatment for patients with multiple myeloma.\(^7\)

The transverse (flexural strength) of a material is a measure of stiffness and resistance to fracture. The transverse strength of poly methylemethacrylate is a very important mechanical property.\(^8\) The incidence of denture fracture might occur both outside and inside the mouth.\(^9\) Outside the mouth, the fracture occurs due to impact if the denture is dropped “impact failure”. Inside the mouth occlusal forces may also cause fracture “fatigue failure” like midline fracture that may occur as a consequence of flexural fatigue which results from cycling deformation of denture base during function\(^9\). The fatigue failure is more common than impact failure.\(^11\)

The transverse strength of HCAR was considerably enhanced by including either metal wires or glass fibers. It has been long hypothesized that the addition of synthetic fibers to the monomer-polymer mixture may strengthen the resultant acrylic resin\(^12\).

Therefore, the present study was undertaking to: Evaluate the effect of incorporation of Calcium salt CaCO\(_3\) from egg shell and Fluoride salt NaF on transverse strength of RESPAL® NF(VEIN 24) heat cured acrylic resin denture base.

**MATERIALS AND METHODS**

Preparation of CaCO\(_3\) powder: All chicken eggshell used in this study is from the local markets. Egg casings were removed for internal crust then cover was lifted lining of the peel and then wash away the chaff very well to be sure to remove the cover lining and dried. white eggshell grind by coffee grinding and purification by Brevettato Seeve (100µm, Bergamo, Italy), to nano particles by adding to 100ml of water and filtering by Whitman filter paper no.6 (wet strength 115, England) slow retention producing fine crystals. Then, dried and sterilized by autoclave 1hr. at 125 °C and finally calcium carbonate crystals white powder produced.\(^3\)

Sodium fluoride: NaF(99.99%) (Aldrich chemical company, USA) powder chemical compound obtained from laboratory of College of science /chemistry department of Mosul university.

Preparation of samples: Preparation of samples by cutting hard elastic foil 2.5mm thickness into plastic Specimens 65mmx-10mmx2.5mm length, width and thickness respectively according to American Dental Association specification no.12,1975, Flasking was done by mixing in ratio of 28 -32 ml of water: 100 gm of stone\(^13\), and poured in lower half of the flask and vibrated. A slurry of stone was applied to one surface of the plastic specimens and placed over the stone in the flask to prevent incorporation of air between the stone and plastic. After the stone was set, a separating medium was applied over the stone, and then the upper half of the flask was placed over and filled with stone (Figure (1)).

![Figure (1): lower half of flask with plastic specimens](image-url)
After complete setting of the stone, the two halves of the flask were opened and the plastic specimens were removed. After drying, a separating medium was applied to both halves of the flask. Packing accomplished by applying the polymer to the monomer of the RESPAL heat cured acrylic resin which was placed in a glass jar and mixed together in a ratio of 3:1 by weight according to the manufacture instruction. The experimental groups of heat cured acrylic resin with additive have been prepared by mixing powder polymer\(^{18}\) 50gm polymer with 1.5gmCaCO\(_3\) for 2.5% and 50 gm polymer with 2.5gm CaCO\(_3\) for 5% and 50gm polymer with 5gm CaCO\(_3\) for 10% with monomer liquid together in glass jar 3:1 by weight, using Sensitive Digital Electrical Balance (A&D Gx-200, Japan). This was repeated for NaF. After the mixture reached dough stage, it was applied in the moulds overfilled and then a polyethylene sheet placed above the acrylic for trial packing. The flask was put under hydraulic press between 1250-2000 psi, and left for 15 minutes before curing\(^{1,14,15}\).

Curing was carried out by placing the clamped flask in the thermostatically controlled water bath for 1 hr at 74°C then 1/2 hr at 100°C, according to the manufacture instruction. After the completion of curing flasks were allowed to bench cool for 30 minutes\(^{16}\) The acrylic specimens were removed from their stone moulds. Then, finished and polished. Three samples with dimensions of 65x10x2.5 ±0.03 mm ADA Specification no. 12, 1975 (Figure (2)) were prepared for each concentration of CaCO\(_3\) and NaF, then the specimens were stored in distilled water at 37°C±1 in the incubator for 48 hrs for conditioning\(^{11}\).

Transverse Strength Measurement Test

The test was applied by using Digital Electronic Force Gauge (Amada, Japan) (Figure (3)) a three points bending on an instron testing.

The device was supplied with a central loading plunger and two supports, with polished cylindrical surfaces of 3.2 mm in diameter and 50 mm between supports. The supports should be parallel to each other and perpendicular to the central line\(^{17}\). The tests were carried out with cross head speed of 5mm/min. The test samples held at each end of the two supports, and the loading plunger placed mid way between
the supports, the samples were deflected until fracture occurred. The transverse strength was calculated using the following equation:\(^{(15)}\):

\[
S = \frac{3PI}{2bd^2}, \text{ N/mm}
\]

\(S\) = transverse strength
\(P\) = maximum force exerted on specimen (N)
\(I\) = distance between supports (mm)
\(b\) = width of specimen (mm)
\(d\) = depth of specimen (mm)

**Statistical Analysis**

The following statistical methods were used to analyze and assess the results via SPSS V. 11.5:

1. Descriptive statistics include mean ± standard deviation values.
2. ANOVA and Duncan multiple range test were used. The statistical results were considered significant at \(p \leq 0.05\).

**RESULTS**

The Mean and Standard deviation of transverse strength for the first group control HCAR (164.400 ± 17.7584 N/mm), the 2nd group HCAR mixed with 2.5% CaCO\(_3\) (123.600 ± 10.3923 N/mm), the 3rd group HCAR mixed with 5% CaCO\(_3\) (121.600 ± 4.5431 N/mm), the 4th group HCAR mixed with 10% CaCO\(_3\) (127.600 ± 22.8421 N/mm) and the 5th group HCAR mixed with 2.5% Na (179.600 ± 19.8756 N/mm), the 6th group HCAR mixed with 5% NaF (152.400 ± 9.0598 N/mm), the 7th group HCAR mixed with 10% NaF (190.000 ± 37.8777 N/mm) were evaluated.

Descriptive statistical analysis of transverse strength for the tested groups were presented in Tables (1 & 2).

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Descriptive statistical analysis of transverse strength for the tested groups were presented in Tables (1 & 2).

Table (1) : Descriptive analysis of Transverse strength for the tested groups (control and CaCO\(_3\))

| T.S    | N   | Mean     | Std. Deviation | Std. Error | Minimum | Maximum |
|--------|-----|----------|----------------|------------|----------|---------|
| Control | 3   | 164.400  | 17.7584        | 10.2528    | 120.286  | 208.514  |
| 2.5%CaCO\(_3\) | 3   | 123.600  | 10.3923        | 6.0000    | 97.784   | 149.416  |
| 5% CaCO\(_3\)  | 3   | 121.600  | 4.5431         | 2.6230    | 110.314  | 132.886  |
| 10% CaCO\(_3\) | 3   | 127.600  | 22.8421        | 13.1879   | 70.857   | 184.343  |
| Total      | 12  | 134.300  | 22.5863        | 6.5201    | 119.949  | 148.651  |

*Different letters mean significant difference.

Table (2) : Descriptive analysis of Transverse strength for the tested groups (Control and NaF)

| T.S    | N   | Mean     | Std. Deviation | Std. Error | 95% Confidence Interval for Mean | Minimum | Maximum |
|--------|-----|----------|----------------|------------|---------------------------------|---------|---------|
| Control | 3   | 164.400  | 17.7584        | 10.2528    | 120.286  to 208.514              | 144.0   | 176.4   |
| 2.5% NaF | 3   | 179.600  | 19.8756        | 11.4752    | 130.226  to 228.974              | 160.8   | 200.4   |
| 5% NaF   | 3   | 152.400  | 9.0598         | 5.2307     | 129.894  to 174.906              | 144.0   | 162.0   |
| 10% NaF  | 3   | 190.000  | 37.8777        | 21.8687    | 95.907   to 284.093              | 146.4   | 214.8   |
| Total    | 12  | 171.600  | 25.0881        | 7.2423     | 155.660  to 187.540              | 144.0   | 214.8   |

One way ANOVA of transverse strength of control and CaCO\(_3\) showed a significance difference at \(P \leq 0.05\) (Table (3)).

Table (3) : one way ANOVA of Transverse strength of control and CaCO\(_3\) (2.5%, 5% and 10%) tested groups

| T.S    | Sum of Squares | df   | Mean Square | F      | Sig.         |
|--------|----------------|------|-------------|--------|--------------|
| Between Groups | 3680.040       | 3    | 1226.680    | 5.081  | .029 *       |
| Within Groups   | 1931.520       | 8    | 241.440     |        |              |
| Total           | 5611.560       | 11   |             |        | *significance at \(P \leq 0.05\)
In comparison with the control, there was a significant decrease at P≤ 0.05 in transverse strength.

One way ANOVA of transverse strength of control and NaF showed no significant differences at P≤ 0.05 (Table (4)) and even with the control.

Table (4) : one way ANOVA of Transverse strength of control and NaF (2.5%,5% and 10%) tested groups

| T.S         | Sum of Squares | df | Mean Square | F    | Sig. |
|-------------|----------------|----|-------------|------|------|
| Between Groups | 2469.120       | 3  | 823.040     | 1.478| .292 |
| Within Groups  | 4454.400       | 8  | 556.800     |      |      |
| Total         | 6923.520       | 11 |             |      |      |

* Not significance at P ≤ 0.05

One way ANOVA of transverse strength of control, NaF and CaCo3 showed significance differences at P ≤ 0.05 (Table (5)).

Table (5) : one way ANOVA of Transverse strength of all tested groups (Control, CaCo3 and NaF)

| T.S         | Sum of Squares | df | Mean Square | F    | Sig. |
|-------------|----------------|----|-------------|------|------|
| Between Groups | 14047.406      | 6  | 2341.234    | 5.695| .004 |
| Within Groups  | 5755.200       | 14 | 411.086     |      |      |
| Total         | 19802.606      | 20 |             |      |      |

*significance at P ≤ 0.05

The transverse strength for the tested groups (Figures (4), (5) and (6)) incorporation of CaCo3 (2.5%,5% and 10%) into acrylic resin will significantly decrease transverse strength at P ≤ 0.05 in comparison with the control. Incorporation of NaF (2.5%, 5% and 10%) into acrylic resin will significantly increase transverse strength in comparison with CaCo3 (2.5%, 5% and 10%). It is not significant with the control.

Figure (4): Mean ±S.D and Duncan Multiple Analysis range test for transverse strength test of tested groups

A. not significance with control.
B. significance with 10%NaF.

Figure (5): Mean ±S.D and Duncan Multiple Analysis range test for transverse strength test of tested groups
The transverse strength of heat cured acrylic resin (HCAR) was considerably enhanced by including either metal wires or glass fibers. The influence of incorporation of Calcium salt CaCo3 from eggshell and Fluoride salt NaF with heat cured acrylic resin has not been tested before.

In our study Figures (4, 5 and 6) showed that adding CaCo3 (2.5%,5% and 10%) to HCAR will significantly decrease transverse strength in comparison with the control HCAR. This was within the acceptable range of ADA specifications ,1975.While adding NaF (2.5%,5% and 10%) to HCAR will significantly increase transverse strength in comparison with CaCo3 (2.5%,5% and 10%) and not significant with the control .

This result agreed with Al-Bahar(2013) who concluded that after adding Hydroxyapatite (2% ,5% ) to heat cured acrylic resin showed an increase in strength of denture base.(18)

This result agreed with the Vodjdani and Kalid (2006) who showed that the transverse strength of heat polymerized denture base resin was considerably enhanced by including either metal wires or glass fibers.(12)

If fibers are to be used to strengthen a polymer material, optimal adhesion between the fibers and the polymer matrix is essential. Impregnation of reinforcing fibers with resin allows fibers to come into contact with the polymer matrix. (19,20)

This can be simulate the incorporation of NaF and CaCo3 into acrylic resin in this study.

This result agreed with Ayad et al. (2008) who found that the transverse strength of Metrocryl high-impact acrylic resin can be increased significantly when reinforced with zirconia in a concentration of 5% and 15% respectively.(21)

**CONCLUSIONS**

It was concluded that adding NaF to HCAR showed an increase in strength of denture base , but significant decrease in strength when adding CaCO3 to HCAR and this within acceptable range of ADA specification no.12 ,1975.

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