The Effectiveness of Aluminum Potassium Sulfate Micro-Particles Addition into Soft Denture Lining Material on Tensile strength and Peel bond Strength of Soft Denture Lining Material

Ali Mohad B.D.S. (1)
Abdalbseet A Fatalla B.D.S., M.Sc., Ph.D. (2)

ABSTRACT
Naturally available products have been used widely for centuries in handling human disease. The present study aimed to determine the effect of aluminum potassium sulfate addition into the soft liner on tensile strength and peel bond strength. The effect of aluminum potassium sulfate evaluated by two methods, first one include incorporation of KAL (SO₄)₂ into soft liner monomer in concentration (2%,3% by wt.) while the second method include immersion of soft liner specimens in solution of KAL(SO₄)₂ in concentration(5%,10% percent) during time periods (0,10 minutes). In conclusions, the results of current study encourage use KAL (SO₄)₂ with in soft liner material.

Keywords: Aluminum Potassium Sulfate, Micro-Particles, Denture Lining Material.

INTRODUCTION
Soft denture lining material used in patient suffering from pain or soreness resulting from tissue contact with hard denture base. Addition of soft denture lining material ensuring optimal adaptation of the denture to the underling tissue. Soft denture lining material characterized by high resiliency so acting as shock absorber reducing load transmission to the underling tissue. Using of soft liners with time becomes more prevalent for providing comfort for patient wearing denture. Soft liners are frequently used for patients who cannot bear wearing a conventional denture base. Aluminum potassium sulfate(alum) having chemical formula KAL(SO₄).12H20 and generally having no odor, no color sold crystal that return white in color in air that used in food preservation and water purification. The alum has been recommended as active ingredient part in mouth wash by the Counter Advisory Panel of U.S. Food and Drug Administration (FDAs).

MATERIALS AND METHODS
The study divided into two parts:
1. Incorporation of KAL (SO₄)₂ into soft liner (2%,3% by wt.). Where it mixed with soft liner monomer.
2. Immersion of soft liner specimens in solution of KAL (SO₄)₂ (5%,10%, for 10 minutes). In addition to control group.

Fifty specimens for each test were made, which then subdivided to five groups.

Specimen preparation:
Tensile strength specimens preparation:
Specimen was prepared, with central cross section area (33*6*3mm) (ASTM D412). The lower portion of the flask was filled with dental stone that mixed according to the manufacturer's instructions (w/p ratio; 20ml/100g). The plastic patterns was invested into the stone mixture, after setting of the stone, the stone surface was coated with separating medium then the upper half of the flask was positioned on the top of the lower half and filled with stone. The flask was well covered and left for stone setting. After 1 hr the flask was opened and the standard specimen was drawn out (Figure1).
The Effectiveness of

Figure 1: mold preparation for tensile strength specimens

**Peel bond strength specimen’s preparation:**
The preparation of the peel bond strength test specimens was made according to ASTM D903-93. Two rectangular stainless steel plates one includes holes with dimensions of 100 x 10 x 2 mm (length, width, height respectively) for PMMA, and the other includes holes with dimensions 150 x 10 x 2 mm (length, width, height respectively) for soft liners (Figure 2). The flask consist of four plates two of them 5 mm in thickness were used as a cover while the others 2 mm in thickness contain holes inside them (6).

Figure 2: Prepared plates for peel bond strength specimens.

**Proportioning and mixing of heat cure acrylic soft liner:**
The liquid mixed with powder according to manufacturer direction (P/L ratio1.2g:1ml) in dry clean glass jar and covered with lid.

**Incorporation of aluminum potassium sulfate into soft liner:**
The weighed amount of KAL(SO₄)₂ added to the soft liner monomer and mixed in clean dry glass jar using probe sonicator until become completely homogenous then soft liner powder added. Keeping in mind to subtract the weight of KAL(SO₄)₂ from weight of soft liner powder.

**Packing packing method for tensile specimens:**
When the soft liner reach to dough stage, it was placed on mold space prepared previously and secured with polyethylene sheet then upper part placed on it and transferred to the hydraulic press to expel the excess soft liner. Then the flasks removed from press, opened then remove polyethylene sheet and excess soft liner. Then the flask closed and transferred to hydraulic press for 5 minutes under pressure (100g/cm²) then clamping the flask (7).

**Packing method for peel strength specimens:**
The first step in specimen preparation include packing of heat cure acrylic resin, this material was proportioned and mixed according to the manufacturer’s instructions, P/L ratio (2.3g/1ml) then inserted into the holes prepared for acrylic in the stainless steel plates (6). The flask was closed and placed under hydraulic press until reach (100 MPa) and left for 5 minutes (7). After that, the specimens were immersed in boiling water for 20 minutes. After polymerization, the flasks were kept for cooling for 30 min followed by cooling under running water for 15 minutes. The acrylic strips were deflasked and trimmed away. The surfaces of acrylic that bonded with soft liners were smoothed using 240-grit silicone carbide paper, cleaned, and dried. Then the acrylic specimens were reflasked (6) (Figure 3).

Figure 3: acrylic specimens in flask.
Part of the acrylic specimen surface of all specimens was covered with a piece of tinfoil to ensure that only 70mm length of the lining material was bonded (8) (Figure 4).

![Figure 4: Application of tinfoil on acrylic specimens.](image)

Then the soft lining material mixed and inserted into the hollow space in the plate designed for soft liner (Figure 5).

![Figure 5: Packing of heat cure soft liner.](image)

This assembly was covered with another plate 5mm thick then the nuts were tightened. The flask was placed under hydraulic press with slow pressure to allow even flow of soft liner dough until reach (100MPa) and left for 5 minutes (7).

**Curing and finishing:**

The packed dental flask immersed in digital water path. Curing time was according to the manufacturer’s instructions (70°C for 90 minutes then for 30 minutes after temperature raising to 100°C) (9). When curing cycle completed, the flask removed and allow to cool for 30 minutes then flask opened and specimens removed from their mold. The access soft liner material removed using sharp blade and finished by fine grit polishing silicon bur and fine grit sand paper.

**Evaluating the effect of aluminum potassium sulfate on tensile strength and peel bond strength of the soft-liner:**

**Tensile strength test procedure:**

Thickness of the test specimens was measured at the center of the test specimen by a vernier caliper with digital readout. The width of the test specimens was also measured to calculate the cross sectional area of the narrow portion of the specimen. The specimen was mounted in a computerized universal testing machine in a way that exposes only the central area of specimens during testing (10). The upper member of the universal testing machine remained fixed, while the lower member moved at a constant rate of (500 mm/min) (11), every specimen was stretched until it cuts. The maximum force at break was then recorded by the computer software. In accordance with ISO 37: 2011 the ultimate tensile strength was calculated from the maximum stretching force at break divided by the original cross sectional area of the narrow portion of the specimen (width × thickness).

**Peel bond strength testing procedure:**

The peel bond strength test was analyzed according to ASTM D903-93 in a universal testing machine at an angle of 180º and speed of 152 mm/min. The non-relined portion of the heat-cured acrylic resin was clamped on the upper clutch of the equipment while the free portion of soft lining
material was folded and fixed in the lower clamp and holding the specimen against an alignment plate (Figure 6).

![Image](https://via.placeholder.com/150)

**Figure 6: Peel bond specimen under testing.**

After the specimens were tested and removed from the testing device, the nature of the bonding failure was evaluated by naked eye, and categorized as cohesive, adhesive or mixed. Cohesive failure refers to tearing within the soft liner material, adhesive failure refers to total separation at the interface between the soft liner and acrylic resin, and mixed failure refers to both. The peel bond strength was calculated by using the following equation:

\[ \text{Peel strength} = \frac{\text{average load}}{\text{width of the sample}} \]

**Results**

**Scanning electron microscope (SEM):**

SEM results of soft liner before and after the addition of 2% and 3% by wt. KAL(SO₄)₂ micro particles powder (Figure 7).

![Image](https://via.placeholder.com/150)

**Figure 7: Scanning electron microscope results: control group (A,B), 2% group of KAL(SO₄)₂ (C,D), 3% group of KAL(SO₄)₂ (E,F).**
Test of Homogeneity of Variances
Before starting with ANOVA table multiple comparisons test, the variances of tested groups within each test were analyzed by running the Levene’s test of homogeneity. According to primary analysis of data homogeneity, Games-Howell test was selected for multiple comparisons of incorporation part and Bonferroni test was selected for multiple comparisons of immersion part of tensile strength and peel strength tests.

**Tensile strength test**
Experimental incorporation group 3% by wt. of KAl (SO₄)₂ showed highest mean value (6.2497 MPa) followed by the experimental group 10% (immersion in KAl (SO₄)₂ solution) with mean value (2.786 MPa) followed by incorporation group 2% with mean value (2.729 MPa) then the experimental immersion group 5% (2.293 MPa) mean value while the lowest mean value was for the control group where the mean value (2.113 MPa).

One-way ANOVA Table for tensile strength test results showed significant difference between tested groups (Table 1 and 2).

**Table 1: One-way ANOVA Table of incorporation part.**

| Sum of Squares | Df  | Mean Square | F   | Sig. |
|---------------|-----|-------------|-----|------|
| Between Groups | 99.624 | 2 | 49.812 | 85.849 | 0.000 |
| Within Groups | 15.666 | 27 | 0.580 |   |   |
| Total         | 115.290 | 29 |   |   |   |

**Table 2: One-way ANOVA Table of immersion part.**

| Sum of Squares | Df  | Mean Square | F   | Sig. |
|---------------|-----|-------------|-----|------|
| Between Groups | 2.428  | 2 | 1.214 | 3.853 | 0.034 |
| Within Groups | 8.508  | 27 | 0.315 |   |   |
| Total         | 10.936 | 29 |   |   |   |

To compare the mean values of tested groups, Games Howell test was conducted for incorporation groups, while Bonfferoni was conducted for immersion groups. There was significant difference between groups except the difference between control group and 5% immersion group and between 5% immersion group and 10% immersion group were non significant (Table 3 and 4).

**Table 3: Games Howell multiple comparisons test of incorporation part results.**

| Mean Difference (I-J) | P value | Sig. |
|-----------------------|---------|------|
| Control 2% 3% 2%       | -0.61600* | 0.034 | S |
| Control 3% 3% 2%       | -4.13670* | 0.000 | HS |
| Control 2% 3% 2%       | -3.52070* | 0.000 | HS |

**Table 4: Bonfferoni multiple comparisons test of immersion part results.**

| Mean Difference (I-J) | P value | Sig. |
|-----------------------|---------|------|
| Control 5% 10% 5%     | -0.18000 | 1.000 | NS |
| Control 10% 10% 5%    | -0.67300* | 0.037 | S |
| Control 5% 10% 5%     | -0.49300 | 0.180 | NS |
Peel bond strength test:
The control group showed highest mean value (1.921 N/mm) followed by experimental immersion group 5% (1.847) mean value, followed by experimental immersion group 10% (1.459) mean value then the experimental incorporation group 2% (1.082) mean value while the lowest mean value was for 3% incorporation group (0.8387). Upon examining the mode of failure of the specimens, it appeared that the specimens of control group failed cohesively. The experimental 0.05 immersion group, 7 specimens show both failures while the other failed cohesively. The experimental 10% immersion group, 8 specimens show both failures while one specimen failed cohesively and the other failed adhesively. In 2% incorporation group all the specimens failed cohesively except one show both failures while in 3% incorporation group 7 specimens show adhesive failure while the other show both failures.

One-way ANOVA Table for peel strength test results showed highly significant difference between all tested groups (Table 5 and 6).

### Table 5: One-way ANOVA Table for peel strength test incorporation results.

| Sum of Squares | Df | Mean Square | F   | Sig. |
|----------------|----|-------------|-----|------|
| Between Groups | 6.448 | 2 | 3.224 | 65.427 | 0.000 |
| Within Groups  | 1.331 | 27 | 0.049 | | |
| Total          | 7.779 | 29 | | | |

### Table 6: One-way ANOVA Table for peel strength test immersion results.

| Sum of Squares | Df | Mean Square | F   | Sig. |
|----------------|----|-------------|-----|------|
| Between Groups | 1.232 | 2 | 0.616 | 7.333 | 0.003 |
| Within Groups  | 2.267 | 27 | 0.084 | | |
| Total          | 3.499 | 29 | | | |

To compare the mean values among study groups, Games Howell test was conducted for incorporation groups, while Bonfferoni was conducted for immersion groups. There was highly significant difference between all groups except the difference between control group and 5% immersion group which was non-significant. (Table 7 and 8).

### Table 7: Games Howell multiple comparisons test of peel strength test incorporation results.

| Incorporation groups | Mean Difference (I-J) | P value | Sig. |
|----------------------|-----------------------|---------|------|
| Control              | 2%                    | 0.83900⁰| 0.000| HS  |
|                      | 3%                    | 1.08230⁰| 0.000| HS  |
|                      | 2%                    | 0.24330⁰| 0.000| HS  |

### Table 8: Bonfferoni multiple comparisons test of peel strength test immersion results.

| Immersion groups    | Mean Difference (I-J) | P value | Sig. |
|---------------------|-----------------------|---------|------|
| Control             | 5 %                   | 0.07400 | 1.000| NS  |
|                      | 10%                   | 0.46200⁰| 0.004| HS  |
|                      | 5%                    | 0.38800⁰| 0.017| S   |
Discussion:
Soft lining materials play a major role in prosthetic dentistry, due to the viscoelastic properties of denture liners which reduce and redistribute the functional load over the denture bearing area (13). Aluminum potassium sulfate is natural products have been used for centuries in treating human diseases and they contain components of therapeutic value. Natural products are environmentally safer, easily available, and cheap (14).

Tensile strength test
The maximum stress a material can withstand before being locally deformed is known as tensile strength (15). Among the several preferable mechanical properties of soft lining material, high tensile strength is of great importance to final prosthesis (16).

The result of this study revealed that significant increase in mean values of experimental groups by using concentration 0.02, 0.03 by wt.(incorporation to the soft liner) and 5%, 10% of KAL(SO₄)₂ solution (immersion of soft liner in the KAL(SO₄)₂ solution), however the highest increase was noticed in 3% by wt. KAL(SO₄)₂ micro-particles concentration. The results were agreed with the results of Waters and Jagger in 1999.

Peel bond strength test
Peel bond strength is the average load per unit width of bond line required to separate bonded materials, when the angle of separation is 180° (12).

The result of this study revealed that there was decrease in peel bond strength of experimental groups with various concentration (2%, 3% by wt. incorporation of KAL (SO₄)₂ to the soft liner and 5% immersion and 10% immersion groups of soft liner specimens in the KAL (SO₄)₂ solution) in comparison with control group, with highest value for control group and the lowest value for 3% by wt. incorporation group.

The KAL (SO₄)₂/polymer bonding have an influence on peel bond property, where stronger KAL (SO₄)₂/polymer bond increases the values of this property and vice versa (11).

The experimental groups showed a tendency to fail adhesively in 20% of specimens (8 specimens of experimental groups) after the addition of the KAL (SO₄)₂ micro-particles, this may be related to the bonding surface swelling due to the absorption of water by the soft denture liner (because of hydrophilic nature of KAL (SO₄)₂) and stress may increase in the interface between soft denture liners and denture base acrylic resin leading to adhesive failure.

In controversy, cohesive failure in control group was predominant, due to its poor tear resistance. In 47.5% of experimental specimens show mixed failure (adhesive and cohesive) this may due to bonding surface swelling and/or decreasing in tear resistance of soft liner after KAL(SO₄)₂ addition.

Reduction in peel bond strength may be due to aggregation of KAL (SO₄)₂ micro-particles because of higher surface energy and this aggregation can cause micro fracture that weaken the polymer structure.

References
1 Aydin AK, Terzioglu H, Akinary AE, Ulubayram k, Hasirei N. Bond strength and failure analysis of lining materials to denture resin. Dent mater. 1999;15(3):211-21.
2 Bulad k, Taylor RL, Verran J, McCord JF. Colonization and penetration of denture soft lining materials by Candida albicans. Dent Mater. 2004;20(2):167-175.
3 Sarac D, Sarac YS, Kurt M, Yuzbasioğlu E. The effectiveness of denture cleansers on soft liners colored by food colorant solutions. J Prosthodont. 2007; 16(3):185-191.
4 Olmez A, Can H, Ayhan H, Olur H. Effect of alum-containing mouthrins in children for plaque and salivary levels of selected oral microflora. J. Clin. Pediat. Dent. 1998;22:335-341.
5 Dootz ER, Koran A, Craig RG. Comparison of the physical properties of 11 soft denture liners. J Prosthet Dent.1992; 67:707-712.
6 Sanchez-Aliaga AS, Pellissari CV, Arrais CA et al. Peel bond strength of soft lining materials with antifungal to a denture base acrylic resin. Dental Mater J. 2016;35(2):194–203.
7 Yassir AD. The effect of addition of zirconium Nano particle on antifungal activity and some properties of soft denture lining material. Journal of Baghdad Collage of Dentistry. 2017; 29(4):27-32.
8 Demir H, Dogan A, Dogan OM, Keskin S, Bolayir G, Soygun K. Peel Bond Strength of Two Silicone Soft Liners to a Heat-cured Denture Base Resin. J Adhesive Dent. 2011;13(6):579-584.
9 Issa MI. Evaluating the effect of silver nanoparticles incorporation on antifungal action and some properties of soft denture lining material. Journal of Baghdad Collage of Dentistry. 2015; 27(2):17-23.
10 Urban VM, de Souza RF, Arrais CA, Borsato KT, Vaz LG. Effect of the association of nystatin with a tissue conditioner on its ultimate tensile strength. J Prosthodont. 2006;15: 295-299.
11 Waters MG, Jagger RG. Mechanical properties of an experimental denture soft lining material. J Dent. 1999; 27: 197-202.
12 Kutay O. Comparison of tensile and peel bond strengths of resilient liners. J Prosthet Dent. 1994; 71: 526-530.

13 Pinto JR, Mesquita MF, Henriques GE, de Arruda NMA. Effect of thermocycling on bond strength and elasticity of 4 long term soft denture liners. J Prosthet Dent. 2002;88: 516–521.

14 Osuala Fl, Ibibio obe MT, Okoh HI, Aina OO, Igbasi UT, Nshogu ME. Evaluation of the efficacy and safety of Potassium Aluminium Tetraoxosulphate in the treatment of tuberculosis. European J of Biol Sci. 2009; 1:10-14.

15 Sakaguchi RL, Powers JM. Craig’s Restorative Dental Materials-E-Book, Elsevier Health Sciences. 2012.

16 Oguz S, Mutluay MM, Dogan OM, et al. Effect of thermocycling on tensile strength and tear resistance of four soft denture liners. Dent Mater J. 2007; 26: 296-302.

الخلاصة:

المواد المتوفرة طبيعياً استخدمت لقرن في علاج أمراض الإنسان. الهدف من الدراسة الحالية هو تحديد تأثير إضافة كبريتات الالمنيوم والبوتاسيوم الى مادة الطقم المرنة على قوة السحب وقوة ترابط التقشير. تأثير إضافة كبريتات الالمنيوم والبوتاسيوم قد تم تقييمه من خلال طريقتين، الطريقة الأولى تتضمن دمج كبريتات الالمنيوم والبوتاسيوم مع سائل مادة الطقم المرنة في تركيز (2,3% بالوزن) بينما الطريقة الثانية تتضمن غمس عينات مادة الطقم المرنة في محلول كبريتات الالمنيوم والبوتاسيوم بتركيز (5%,10%) خلال فترة زمنية (0,10 دقائق). في الاستنتاج، نتائج الدراسة الحالية تشجع استخدام كبريتات الالمنيوم والبوتاسيوم مع مادة الطقم المرنة.