The Effect of Incorporation Kappa-Carrageenan Powder on the Physical and Mechanical Properties of the Heat-Cured Acrylic-based Soft Denture Lining Material in Clinical Use

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Abstract

Background: Soft denture relining materials have been introduced to prosthetic dentistry since long time to improve the patient's acceptance to dental prosthesis. However, with the growing need to provide the patients with dentures that have improved comfort and masticatory efficiency, there is a growing demand to improve several features of the denture lining materials. Therefore, the aims of this study was to evaluate the effect of incorporation kappa-carrageenan powder as antifungal agent on the hardness and peel bond strength of the heat-cured acrylic-based soft denture lining material.

Materials and Methods: Two percentages of kappa-carrageenan powder (1.5wt.% and 2wt.%) have been selected based on the pilot study that produced the best antifungal effect. A total of sixty samples were prepared and divided into three equal groups groupA:0.0wt.%, groupB:1.5wt.%, groupC:2wt.% of kappa-carrageenan powder additive. Each group was further subdivided into two equal groups for shore-A hardness test and peel bond strength test. Fourier transform infrared spectroscopy (FTIR) analysis was performed to investigate if there is any chemical reaction between the soft lining material and the kappa-carrageenan powder, scanning electron microscope (SEM) was used to determine the distribution of kappa-carrageenan powder in the soft liner matrix.

Results: All the resulted data were analysed using "one-way analysis of variance (ANOVA) and Dunnet T3 post-hoc test at a significance level of $p<0.05$". The result of shore-A hardness test revealed significant increase ($P<0.05$) in the values of hardness of both experimental groups (B and C) when compared with the control group(A). Peel bond strength test revealed non-significant decrease ($P>0.05$) in the values of peel bond strength for experimental group(B), while there was significant decrease ($P<0.05$) for the experimental group(C) when compared to the control group. FTIR analysis showed no difference in the spectra between control group and experimental group this means that there is no chemical interaction between the two materials. SEM test results showed a degree of agglomeration of the kappa-carrageenan powder into the soft liner matrix.

Conclusion: Adding kappa-carrageenan powder to the soft liner affected the physical and mechanical properties of the lining material and the most adverse effect was noted for the group C(2 wt.%) but all effects was in the acceptable range for clinical use.

Keywords: *soft denture lining material; clinical use; dental prosthesis; peel bond strength*

Introduction

Denture soft lining materials have been used in dentistry for many years, the purpose for their use is to

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improve mastication through providing more favorable relation between the denture bearing mucosa and the denture base which will result in more favorable distribution of the occlusal forces. For this reason the denture lining material must exhibit certain properties that allows it to serve properly inside the patient's mouth[1].

The physical and the mechanical properties of the heat-cured acrylic-based and silicone-based soft liners change with time inside the patient's mouth as a result of leaching out the plasticizer[2,3]. Therefore, frequent evaluation and replacement of the used soft lining material is required.

Peel bond strength is one of the methods used to assess the reliability of bond between the denture base and the soft lining material which is thought to closely stimulate the force applied at the interface between the resilient denture liner and the denture base[4]. The results of this test are affected by the thickness and compliance of the material; along with bonding failure predominantly being cohesive within the tested materials[5].

Hardness is another property that is commonly used and measured to assess the quality of different soft liners. Hardness has been defined as the material resistance to indentation[6]. It is one of the most important tests for rubbery materials, and it represents a method for determining the modulus of elasticity since rigid material cannot be used as soft liners[7]. One of the most important advantages of soft lining materials is their viscoelastic properties, which result in significant improvement in masticatory function. Regarding soft acrylic liners this property change with time due to leaching out of plasticizer[4].

Multiple studies have been conducted to improve the properties of the soft lining material, one particular aspect of these studies is the introduction of natural herbal products to the soft lining material to enhance their antimicrobial, physical and mechanical properties. Kappa-carrageenan (κ-carrageenan) powder is a natural sulphated poly saccharide extracted from the red marine algae. It has been identified as possessing high anti-coagulant, anti-oxidant, anti-tumour, and anti-microbial activity[8]. In addition, carrageenan has been used for many years as a food additive and in pharmaceutical applications due to its stabilizing, thickening, and emulsifying properties, and it is accepted by the U.S. Food and Drug Administration (FDA)[9] and the World Health Organization (WHO)[10].

κ-carrageenan powder has been evaluated for its antifungal activity through a pilot study when incorporated into soft lining material as an additive and the two percentages of κ-carrageenan (1.5 and 2wt.%) incorporated into the heat-cured acrylic-based soft liner were proved to have potent antifungal effect. The purpose of this study was to evaluate the effect of each of these percentages on the hardness and peel bond strength of the soft lining material.

Materials and Methods

Samples grouping

A total sixty samples were prepared and divided into two groups of thirty samples for each test (shore A hardness and peel bond strength tests) each group was then subdivided into three groups of ten samples representing the control groupA:0.0wt% and experimental groupsB:1.5wt.% and groupC:2 wt.% of κ-carrageenan powder.

Fourier transform infrared spectroscopy analysis

FTIR analysis was performed using Fourier transform infrared spectroscopy (SHIMADZU/ FTIR.8400,Japan) to investigate if there is any chemical reaction between the soft lining material and the kappa-carrageenan powder. A sample of a pure soft lining material without an additive (0wt.%) and sample with κ-carrageenan powder additive (2wt.%) were cut into small thin flushes and were examined.

Scanning electron microscope

SEM test was preformed using scanning electron microscope (Tescan,Czech Republic) to determine the distribution of κ -carrageenan powder in the soft liner matrix. Three samples were tested by scanning electron microscope, one of the samples represented the control sampleA:0.0wt.% and the other two represented the experimental samplesB:1.5wt.% and C:2wt.%.

Samples preparation:

For both tests samples of the control group were prepared according to manufacturer instructions for the heat-cured, acrylic-based soft liner (Vertex,Netherland) (powder/liquid ratio:1.2g powder/1ml monomer), and mixed together using a clean glass container. For experimental samples, the weight of the kappa-carrageenan powder (Sigma al-drich,Denmark) was subtracted from the weight of the soft liner powder to obtain an accurate powder/liquid ratio. An amalgamator device (Perfection Plus,United Kingdom) was used to mix the two powders for 40seconds to obtain a homogeneous mixture[11].

Shore-A hardness test: A plastic disks shaped molds with the dimension of (35X6mm) in dimeter and thickness respectively were prepared according to the **ISO-10139-2(2016)** specification[12]. These molds were invested in the lower portion of the dental flask with freshly mixed dental stone (Zermack, Italy) and the flasking procedure was completed in similar way to conventional complete denture construction. All samples were cured according to manufacturer instructions using a thermostatically controlled water bath (Lab.tech., Korea) heated to 70℃ for 90minutes; temperature was then raised to 100℃ for 30minutes. After processing, samples were finished with sharp scissors and polished with a fine-grit, silicone polishing bur under continuous water cooling[11]. All samples were stored in distilled water for 24hours at 37℃ before being tested to eliminate any residual monomer[13].

Peel bond strength test: Samples were prepared according to the **ASTM-D903-93**[14] however the size of the samples was too long so half the measurements was thought to be sufficient[15]. A costume made stainless steel flask was used to prepare the samples, it consisted of four plates with dimensions (260X200mm) length and width respectively two of them was used as covers while the other two consist of slits in which the PMMA and the soft liner material were packed. The thickness of these two plates was (2mm) and the dimensions of the slits of the plate used to pack the PMMA were (100x10x2mm) representing the length, width and height, while the plate used for the soft liner material has slits with dimensions (150x10x2mm) representing the length, width and height respectively[16](Fig.1).

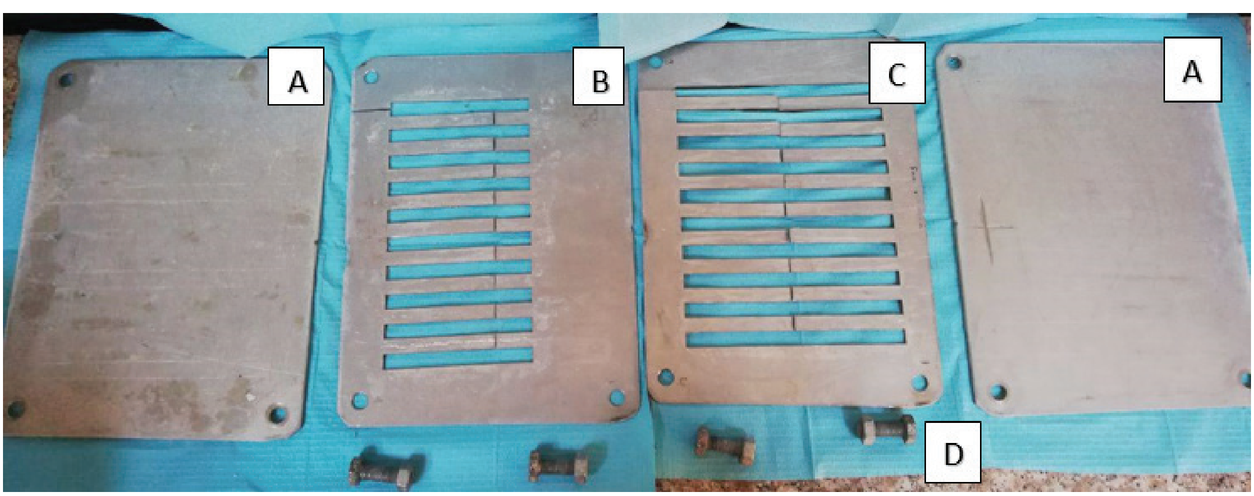


Figure (1) Peel bond strength flask. A) The flask covers, B) Flask plate for poly methyl methacrylate, C) Flask plate for soft liner D) Screws to hold the four plates in tight metal to metal contact.

Samples fabrication included first packing the PMMA material (Vertex, Netherlands) in the flasks which was proportioned and mixed according to manufacturer instructions(P/L2.3g powder to 1ml of liquid). The flask was then closed and a slow pressure was applied using hydraulic press until(100MPa) was reached and left for about 5min. and then following the manufacturer instruction the flask was placed in the water bath for 20min. at 100℃. Following this step the flasks were left to cool for about 30min. and then cooled under running water for about 15min. then the acrylic strips were deflasked, trimmed and finished then reflasked again.

Testing procedure

Shore A-hardness test: shore-A durometer (A081FJ702020, China) was used, five readings at different points on the sample were marked, four points were marked away from the edge in about 5mm and one point in the center, the penetration time was 5sec. and the distance from the indenter was 20mm. The average of the five readings was recorded and determined the value of the shore-A hardness.

Peel bond strength test: The test was accomplished using universal testing machine (WDW-20, China) according to **ASTM-D903-93** specificatios[(14)]{at an angle(180°) and speed (152mm/min)}. The part of the heat cured acrylic resin that was not bonded to the soft lining material was hold by the upper clutch of the machine while the unbonded part of the soft lining material was hold by the lower clutch with the use of an alignment plate to keep the sample in its proper position.

The nature of failure was evaluated with the naked eyes and categorized as either adhesive, cohesive or mixed failure. Peel bond strength value was calculated with the following equation in which the peeling angle was considered 180°.

(Peel bond strength= average load /width of the sample)

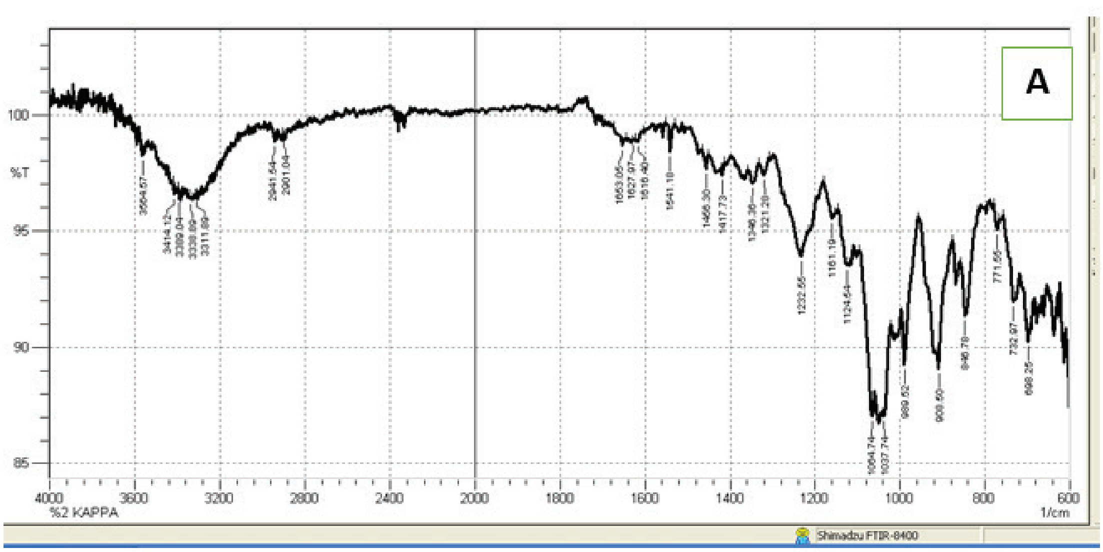
Statistical analysis

Results of the presented research were analysed using SPSS “version 24 computer software”. The descriptive statistics that have been made, includes “means and standard deviation”. “Inferential statistics” were also made, including one-way analysis of variance (ANOVA), for comparison means among all groups, and Dunnet T3 multiple comparison tests, which shows the significance between each of the two different groups, with $p<0.05$ considered significant.

Results and Discussion:

Fourier transform infrared analysis

The results of the FTIR analysis showed there was no differences in the pattern and the alignment of the absorption peaks between the control and the experimental samples. Which means there was no chemical reaction between the soft lining material and the κ-carrageenan powder (Fig.2).



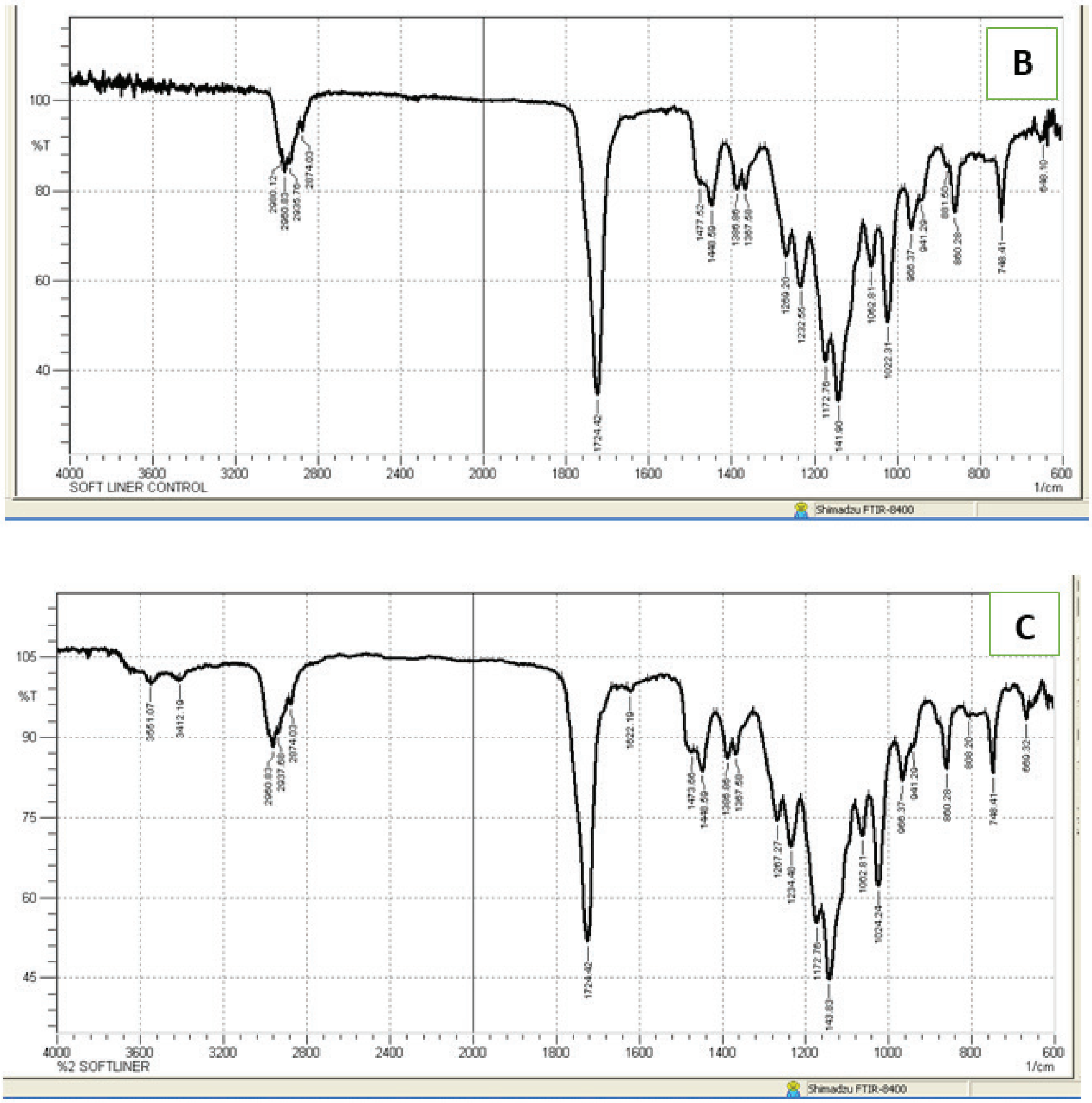


Figure (2): Fourier transform infrared spectroscopy analysis, A) Kappa-carrageenan powder; B) Control sample; C) Experimental sample (2wt.%) kappa-carrageenan powder

Scanning electron microscope test

The results of the SEM test shows there was a degree of agglomeration in the experimental groups which tend to increase with the increase in the kappa-carrageenan powder percentage in the soft lining material (Fig.3).

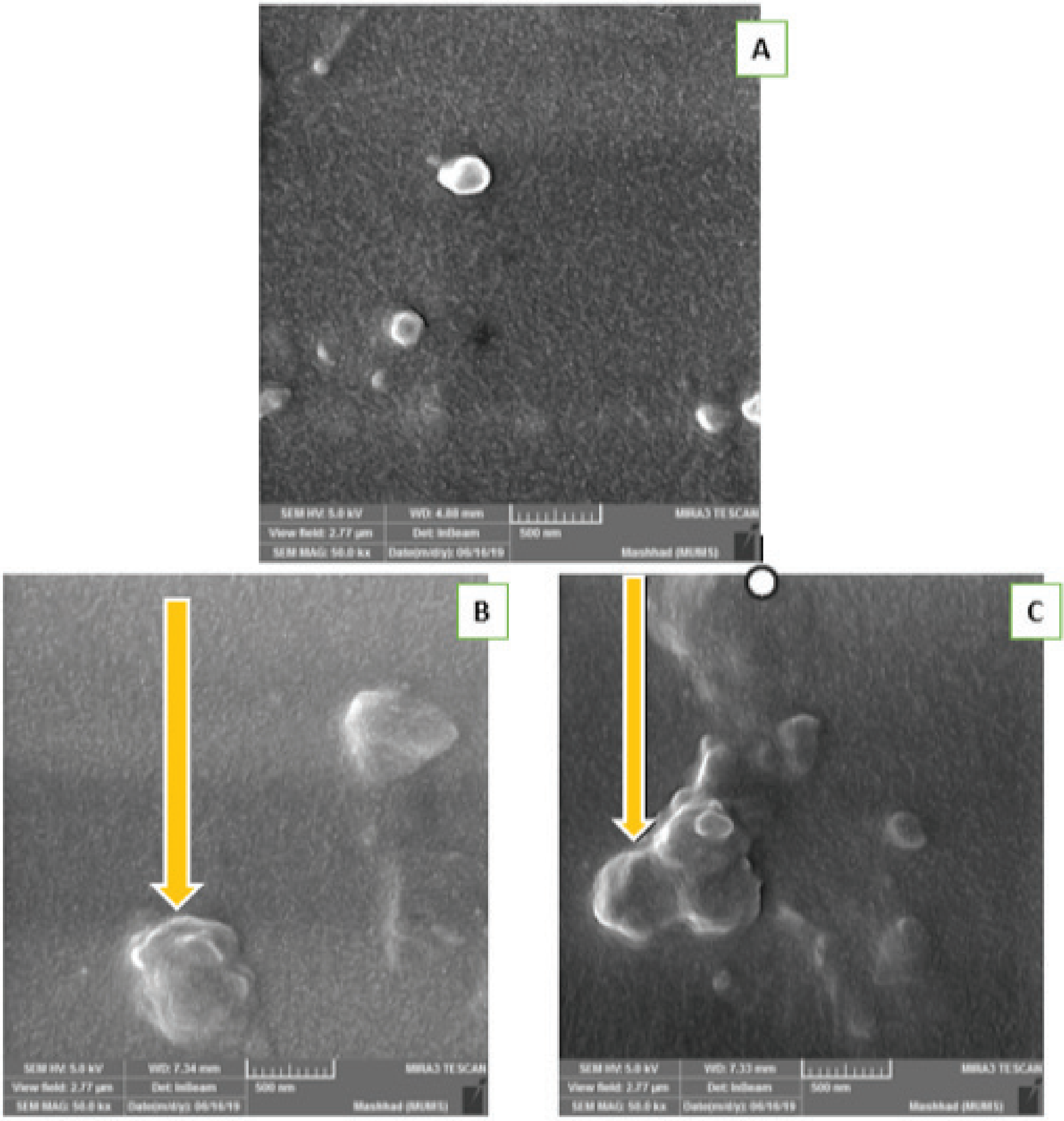


Figure (3): Scanning electron microscope (magnification power 500nm). A) Soft liner control sample without the additive, B) Sample with 1.5wt. % of kappa-carrageenan powder, C) Sample with 2wt. % of kappa-carrageenan powder. Arrows shows the agglomeration of the powder.

Shore-A hardness test

The results of shore-A hardness test showed there were increase in the values of hardness for both experimental groups when compared to the control group, One-way ANOVA showed significant difference of hardness among groups (A, B and C) at $P<0.05$. Dunnett T3 post hoc test showed there was significant difference ($P<0.05$) between the control group and the two experimental groups (B and C) (Table1and2).

Table (1): Mean values of shore-A hardness, peel bond strength tests and ANOVA test.

groups	No. of samples	Shore A hardness				Peel bond strength test			
		Mean	SD	Min.	Max.	Mean	SD	Min.	Max.
A	10	52.58	.817	50.76	53.60	4.630	.600	3.90	5.40
B	10	55.18	1.820	53.40	59.20	4.390	1.030	3.10	5.80
C	10	56.40	.596	55.20	57.20	3.100	.514	2.30	3.80
ANOVA	F-test	26.235				12.051			
	p-value	.000[S]				.000[S]			

Table (2): Multiple comparisons of shore-A hardness and peel bond strength tests between groups using Dunnett T3 post hoc test.

Experimental groups		Shore A hardness		Peel bond strength test	
		Mean diff.	Sig.	Mean diff.	Sig.
A	B	-2.592	.004	.240	.891
	C	-3.812	.000	1.530	.000
B	C	-1.220	.183	1.290	.010

The incorporation of κ-carrageenan powder may altered the polymeric chain penetration by the plasticizer and there by affected its ability for softened gel formation. Additionally κ-carrageenan may acted as a filler that which resulted in increase in the hardness of the soft lining material[17].

The nature of the added material may also influence its effect on the hardness of the soft lining material for instance the increase in hardness that was obtained from this study can be related to the gelatinous nature of the kappa-carrageenan and the formation of agglomerations that was noticed in the SEM results.

Peel bond strength test

The results of peel bond strength test showed both experimental groups (B and C) have decrease values of peel bond strength when compared to the control group. One-way ANOVA for peel bond strength test showed significant difference among the tested groups ($P<0.05$). Dunnett T3 post hoc test showed there was non-significant difference between A and B groups ($P>0.05$), while a significant difference exist between groupA and C ($P<0.05$) (Table1 and Table2).

The mode of failure was non-significant for all the experimental groups. The cohesive failure may be associated with the presence of the κ-carrageenan within the polymeric matrix of the soft lining material while the adhesive failure may be related to the hydrophilic nature of the κ-carrageenan which may absorbed water and decreased the peel bond strength values and caused the samples to fail adhesively (Table3)[16].

Table (3): Fisher exact probability test to compare the mode of failure between the experimental groups.

			Groups			FEPT	P value	Total
			A	B	C			
	Adhesive	N.	0	0	2	6.912	0.105[NS]	2
		%	.00	.00	20.00			6.67
	Cohesive	N.	4	3	0			7
		%	40.00	30.00	.00			23.33
	Both	N.	6	7	8			21
		%	60.00	70.00	80.00			70.00

Ethical Clearance: The Research Ethical Committee at scientific research by ethical approval of both environmental and health and higher education and scientific research ministries in Iraq

Conflict of Interest: The authors declare that they have no conflict of interest.

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