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Effect of Addition of Calcium Carbonate Nano particles on Some Properties of Room Temperature Vulcanized Maxillofacial Silicone Elastomer After Artificial Aging

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Abstract

When a silicone maxillofacial prosthesis is no longer clinically useful after 1.5 to 2 years, a new prosthesis should be made. Adding some kinds of nano-oxide fillers could enhance the situation and address this issue.

This study sought to determine how the CaCo3 nanofiller affected the maxillo facial tear strength of Silicone elastomer RTV50F after artificial aging for 200 hours. Eighty samples in total were split into two groups, each of which having 40 samples, which are then subdivided into four groups: the control group, 0.5%, 0.75%, and 1%. Samples in the prior concentration groups were subjected to rip strength tests both before and after 200 hours of artificial aging. ANOVA and Tukey's HSD tests were used to analyze the data and determine the differences among groupings. Scanning electron microscopy (SEM) and Fourier-transform infrared (FTIR) analysis also used. The ANOVA test show Both before and after aging, the silicone elastomer's mechanical quality (tear strength) was enhanced by the inclusion of $CaCo_3$ nano-particles. The best results were obtained at a concentration of 1% $CaCo_3$ (D group), the efficiency of $CaCo_3$ in reinforcing silicone is supported by these data. The results showed that the addition of CaCo3 to VST-50F silicone showed significant difference compared to the control group. Finally, one can draw a conclusion stating that the addition of calcium carbonate ($CaCo_3$) nano particles as filler improves and increase the tear strength of silicone elastomer both before and after being artificially aged for 200 hours.

Introduction:

Maxillofacial prosthesis has revolutionized prosthodontics by offering solutions for face mutilations brought on by trauma, acquired surgical defects, and congenital abnormalities(1). This rehabilitation serves to not only restore their natural appearance but also enhance their self-image, ultimately contributing to improved psychological well-being(2). However, the mechanical characteristics of silicone elastomers are far from optimal; certain characteristics, such as their short service life and low tensile and tear strength, require improvement. By incorporating fillers, colors, and other additives, an elastic material with improved mechanical and physical qualities can be produced (3) .The impact of different reinforcements on the mechanical characteristics of a silicone elastomer used to prepare maxillofacial prosthetic compositions has been assessed (4). Nanotechnology used in dentistry was in term of 'Nano dentistry'.Itcomprises of diagnostic tools and therapeutic that used nanomaterials(5).To develop a new class of polymeric materials that combine the strength of nano-oxides with the flexibility of organic polymers, research has been focused on creating a novel industrial technique that integrates nanoparticles into a polymeric matrix. (6). In order to obtain the desired outcomes Nanotechnology uses methods to manipulate elements' size, shape, and texture at the required nanoscale. Previously, it was mostly utilized in the fields of physics and chemical manufacturing processes, but new technologies have changed this (7). Due of the procedure's great economic worth and the nano-scale, ultra-fine solid structure of $CaCo_3$, nano-$CaCo_3$ modification has been gaining significant traction among modification techniques. Nano-$CaCo_3$ is crucial for materials' toughening and reinforcement(8). Excellent biocompatibility and minimal toxicity to mammalian cells and tissues are reported for CaCo3 micro- and nanoparticles (9).The consistency of the silicone must be the same as the texture of the skin of the bodily part that has to be repaired(10). According to (11) adding CaCo3NPs to a heat-cured acrylic-based soft denture liner material may enhance its tensile bond strength, tear strength, as the nanoparticle concentration rises Therefore, This study set out to investigate the effects of adding $CaCo_3$NPs on the maxillofacial silicone's tear strength following 200 hours of artificial aging

Materials and Methods:

The type of maxillofacial silicone used in this investigation was VST 50F room temperature vulcanized silicone (TECHNOVENT limited UK) (20–30°C). It was completed with vinyl RTV silicone after being catalyzed in two processes.
in compliance with the manufacturer's instructions, which are met by using the nanofiller CaCo3 powder (The materiales science company USA) and mixing the base (part A) with the cross-linking agent (part B) in a weight ratio of 10:1.

Samples groups:

The total number of samples is 80, divided into two groups, each group has 40 samples, and these 40 are divided into four secondary groups: a control group and three experimental groups containing 0.5%, 0.75%, and 1% weight concentrations of calcium carbonate, every subgroup was tested for tear strength both before and after 200 hours of artificial aging. The VST-50F RTV maxillofacial silicone was supplemented with calcium carbonate at the previously specified weight concentrations figure (1)

Samples' fabrication

After using auto CAD software for design, a CNC machine was used to make a custom plastic mold for the hardness test. In accordance with ISO 619-1, specimens measuring 39.5 mm in length, 16.5 mm in width, and 3 mm in thickness were made using CAD and CNC. The tension concentration location at the 90° angle is where tears should start. apex figure (2). The molds were prepared to pour the maxillofacial silicone and were used for testing both before and after artificial aging (3).

Samples' pouring:

A Chinese digital electronic balance with a 0.000 precision was used for the measurement. To produce the control group specimens, the silicone foundation was first measured according to the manufacturer's instructions and then combined with the accelerator at a 10:1 ratio. Calcium carbonate nanoparticles were added to 100 grams of silicone base (before catalyst addition) at weight percentages of 0.5%, 0.75%, and 1% to create the silicone-nanoparticle mixes. The weight of the nanoparticles corresponding to each concentration was determined as follows:

0.5% = 0.5 g of nanoparticles plus 99.5 g of silicone base.

The silicone base and nano-fillers were mixed using a vacuum mixer at 360 rpm for ten minutes. The first three minutes of mixing were performed without vacuum to prevent premature suction of the nanoparticles. The remaining seven minutes were conducted under vacuum conditions until the pressure reached -10 bar to ensure homogenous dispersion(12). Following this preliminary mixing, the mixture was let to settle and cool for about five minutes at room temperature. After adding the catalyst, the entire mixture was vacuum-mixed once again for five minutes at 140 ±10 rpm at a vacuum pressure of -28 inHg (9).

Pouring the silicone mixture into the sample molds:

After cleaning the plastic mold, the silicone mixture was poured under closely watched conditions, maintaining the temperature at 23 ± 2°C and the relative humidity at roughly 50±10%. The uniform fluid was carefully poured into a disposable plastic syringe. Figure 2.3. The extra mixture was poured straight from the syringe onto the acrylic mold, which was set on a low-speed dental vibrator. After that, the cover was carefully positioned over the material that had been poured into the mold, beginning with one edge and elevating the opposite margin. The homogenous silicone mixture was poured into the mold. To guarantee homogeneous, bubble-free samples, hand pressure was used to remove extra material and air bubbles, and then screws, nuts, and G-clamps were tightened Figure (4-6)

Figure (6): filling the mold with silicone mixture, Hand pressure was applied to release excess material and air bubbles followed by tightening with screws, nuts, and G-clamps

Accelerate artificial aging of specimens:

The specimens were artificially aged for 200 hours in a Weather Ometer device (QUV) with a 12-hour aging cycle in compliance with ASTM G-154/7. The light cycle lasted eight hours at 60 °C, 340 nm, and 1.55 W/m2 of irradiance. There was a 3.45-hour condensation period at 50 °C following a 4-hour dark cycle at roughly 40 nm, 1.55 W/m2 of irradiance, and 15 minutes of water spraying for thermal shock induction. Every 200 hours of artificial aging was equal to around six months of clinical use (real-life aging)(13)

Mechanical Testing Procedures:

Tear strength test:

The testing process was carried out in compliance with the guidelines provided by (8). The thickness of the tear test sample was measured with a caliper close to the center, at three points along the breadth where the fracture is anticipated to occur (close to the right angle). The slit or apex had to be the location of one of the measurements. To calculate the test results, the mean value was noted. The samples were positioned 30±0.5 mm apart, perpendicular to the machine's long axis, and equally spaced from the center. This allowed the samples to be symmetrically attached by the machine's grips in axial alignment with the pull direction (9). It guaranteed that the force was distributed evenly throughout the sample (Figure 2-5). Test samples were stored in a vaccine storage box (Polar Bag, China) at 23±2 °C and 50±5% relative humidity for at least 16 hours before testing. After 200 hours of artificial aging, silicone elastomer VST50F was produced for tear strength testing with a three percent caCo3 addition.

Type C specimens were tested using a universal testing equipment in compliance with (10). The specimens were stretched at a rate of 500 mm/min until the apex

completely broke. The following formula was used to determine tear strength:
Tear strength is equal to f/d,
where d is the specimen's average thickness (m) and f is the maximum force for complete rupturing that has been measured (KN) (11) Figure (6).

Results:

As demonstrated in tables 1,2 and 3, a highly significant difference between all examined groups was found by the ANOVA test. both before and after age, the silicone elastomer's tear strength was greatly increased by the inclusion of $CaCo_3$ nanoparticles. At 1% concentration, the greatest boost was seen. $CaCo_3$'s reinforcing impact on the material was confirmed by the huge effect sizes and statistically significant ($P < 0.001$) nature of all the differences. When Nano Calcium Carbonate ($CaCo_3$) was added to medical silicone Tables (1-4) and Figure (9)
The FTIR spectrum displayed new, distinctive peaks that may indicate surface interactions or functional changes. The primary silicone structure is unaltered, suggesting that the composite was successfully formed without deteriorating. When the concentration of $CaCo_3$ nanoparticles in the silicone matrix was low, the SEM test revealed a good dispersion of the particles. Additionally, the SEM was taken 200 hours after accelerated artificial aging to confirm the particle distributions. As the nano content increases, so does the probability of particle agglomeration within the silicone matrix as shown in figure (10)
The CaCo3 nanoparticles were well distributed throughout the silicone matrix, according to SEM examination. Furthermore, to confirm the particle distributions, SEM was acquired 200 hours following accelerated artificial aging. This is because the concentration of nanofiller increases the probability of particle agglomeration within the silicone matrix after aging.

Discussion:

One of the most popular and reasonably priced RTV silicone elastomers is silicone rubber (VST-50). The demands of everyday prosthetic wear cannot be supported by any maxillofacial material now on the market. To obtain the necessary mechanical strength of a silicone polymeric material, reinforcement with a particular quantity of fiber is needed (11). For both experimental groups, tear strength the material's capacity to withstand tearing pressures acting perpendicular to the surface flaw—increased when in contrast to the control cohorts. By developing Nanoparticles in three-dimensional networks within the polymer matrix can physically trap certain polymer chains. The interaction between the nanoparticles and the polymer matrix can stop the polymer chain from moving against the nanoparticles and against each other, increasing the density and tear strength (14). Additionally, the strain energy during propagation can be distributed using rubber materials (15). This is because, as the tearing process progresses, nanofillers will disperse the energy within the polymer matrix, making it more resistant to tearing and necessitating a higher force and load to rupture fully. Furthermore, the strain energy will be dispersed by the polymer in and around the growing fractures, which could account for the increase in tear strength (16).The final material's mechanical and physical properties can have been influenced by the fillers' concentration, size, and loading. The statistical analysis's findings show that adding $CaCo_3$ nanoparticles to the tested silicone elastomer significantly increased its tear strength ($P \leq 0.001$), with the experimental group's (D) values being noticeably higher than those of the other study groups Table (1). This enhancement might be the result of increased nanoscale surface area, which improved the polymer's adsorption(17). When compared to other experimental groups, the mixed nanocomposites in Group D seem to contain more agglomerates, which could be the result of the $CaCo_3$'s concentration increasing. The findings regarding the tear results following weathering are consistent with (18), who examined the effects of outdoor weathering conditions on TechSil S 25 silicone. Their findings showed a notable decrease in tear readings following weathering. Although the result of this study showed disagreement with many types of research that showed decreased tear strength and increased in the hardness values like(19, 20)

Conclusion:

Adding $CaCo_3$ nanoparticles to VST50F maxillofacial silicone, especially at 1% concentration, significantly increases the maxillofacial silicone elastomers' tear strength and lessens the effects after 200 hours of artifacial aging.

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Conflicts of Interest
The authors declare that they have no conflict of interest

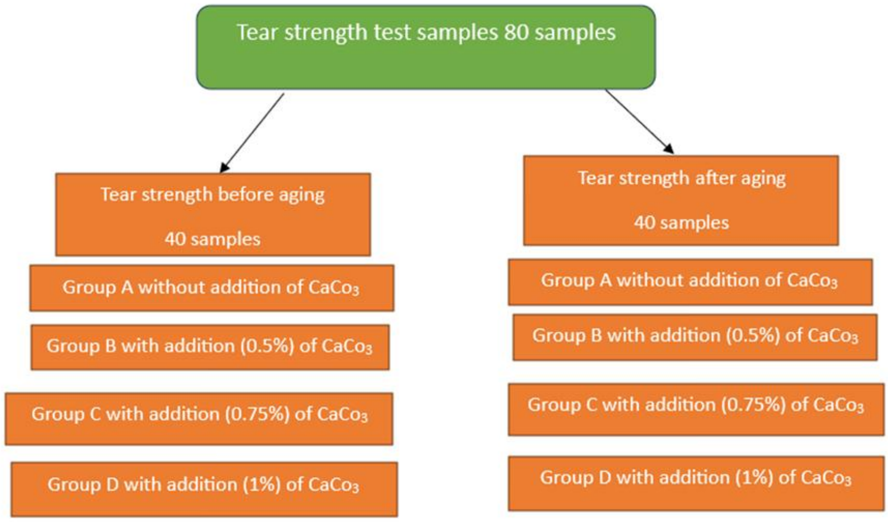

Figure (1): samples grouping

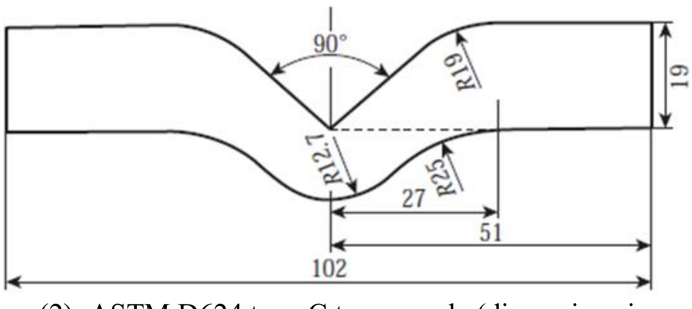

Figure (2): ASTM D624 type C tear sample (dimensions in mm).

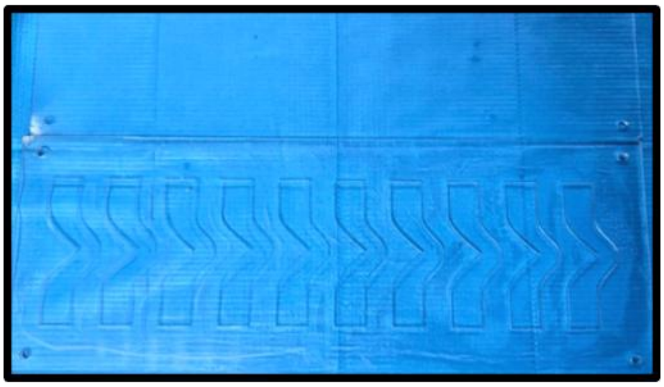

Figure (3): tear strength mold

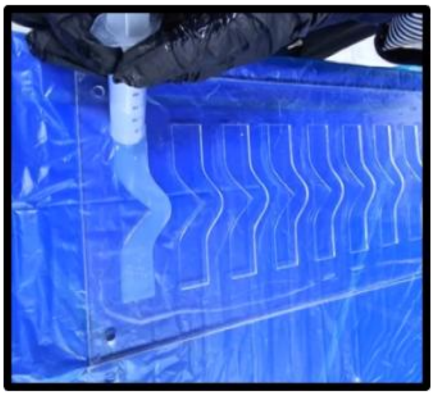

Figure (4): disposable plastic syringe was gently filled with the homogenous mixture and pouring the mold

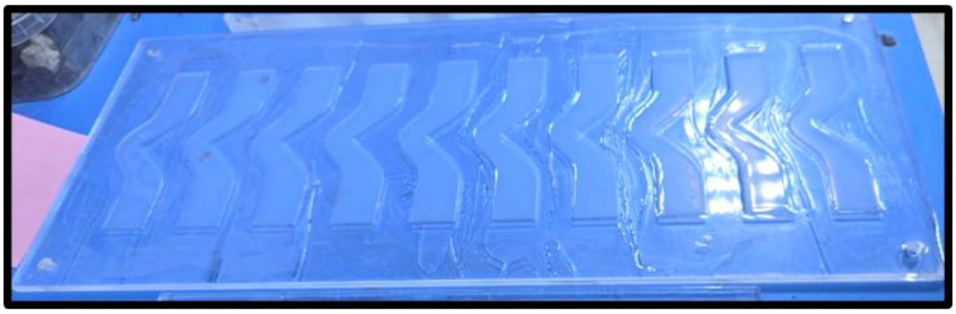

Figure (5): the mold filled with silicone elastomer and cover it

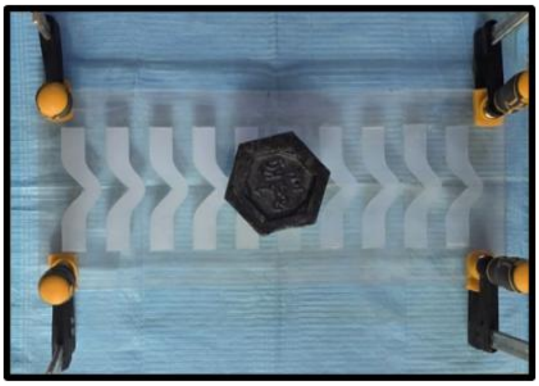

Figure (6): filling the mold with silicone mixture, Hand pressure was applied to release excess material and air bubbles followed by tightening with screws, nuts, and G-clamps

Figure (7): tear strength specimen during mechanical measuring

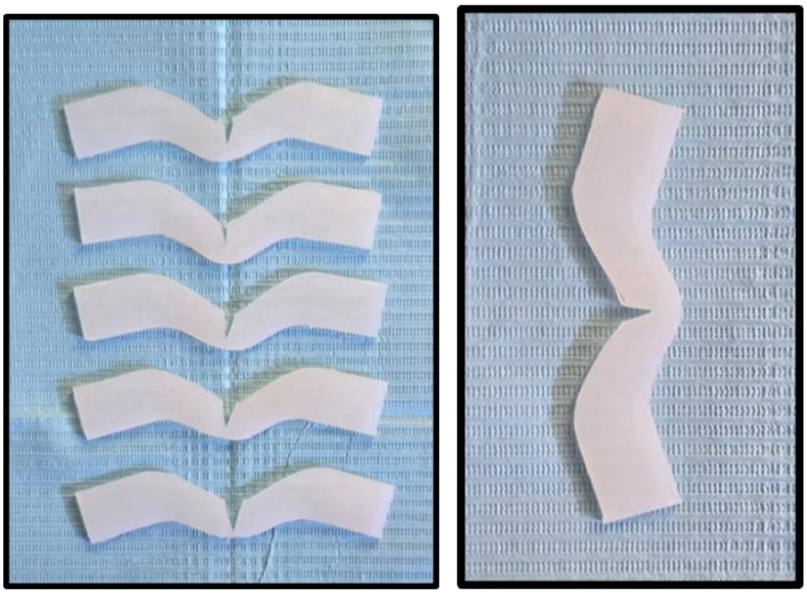

Figure (8): Tear strength sample after being broken

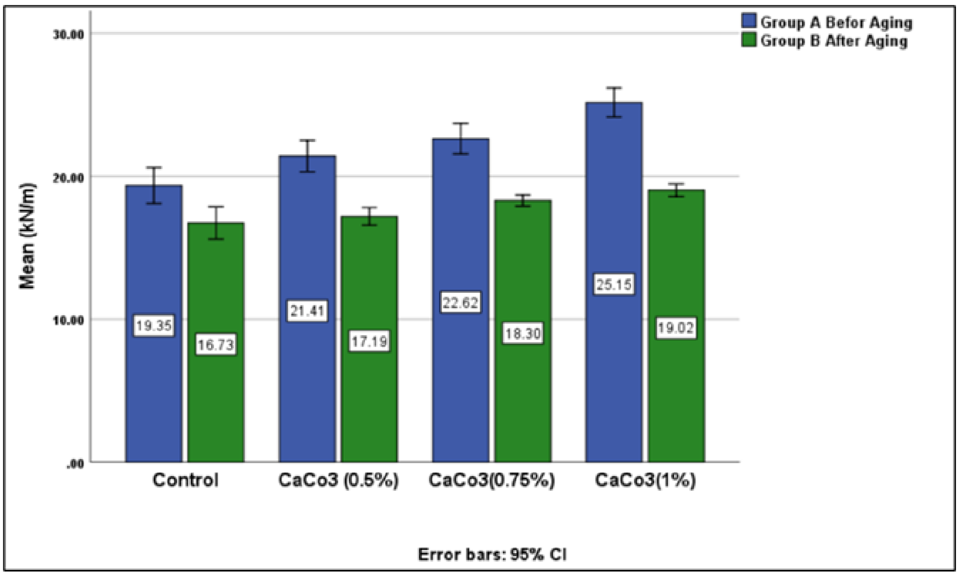

Figure (9): The bar chart shows the mean and standard deviation of the Tear strength values before and after artificial aging

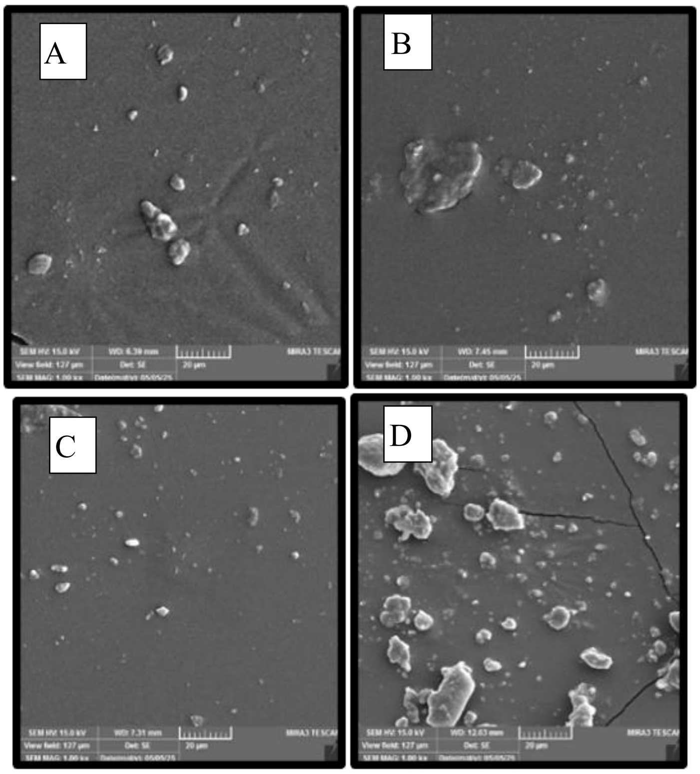

Figure (10): SEM of VST50F maxillofacial silicone
A,20um magnification with the addition of nano-Fillers (0.5%) before aging.
B, 20um magnification after aging. C,20 um magnification with the addition of nano Fillers (1%). D, 20um magnification after aging

Table (1). Descriptive and inferential statistics of the tear strength before and after aging among group

Variables	*Groups*	*Mini*	*Max*	*Mean*	*SD*	*F*	*P-value*	*ES*
G1 Tear strength before aging	**Control**	16.32	21.70	19.35	1.76	23.953	<0.001 HS	0.816
	$CaCo_3$ (0.5%)	18.60	23.95	21.41	1.54			
	$CaCo_3$ (0.75)	19.75	24.37	22.62	1.49			
	$CaCo_3$ (1%)	22.30	27.00	25.15	1.42			
G2 Tear strength after aging	**Control**	14.70	19.50	16.73	1.58	11.089	<0.001 HS	0.911
	$CaCo_3$ (0.5%)	16.00	18.90	17.18	0.86			
	$CaCo_3$ (0.75)	17.50	19.30	18.29	0.54			
	$CaCo_3$ (1%)	18.00	19.80	19.02	0.61			

Table (2):Tukey's HSD of Tear strength test before and after aging between groups.

Groups	*Studied groups*	*No.*	*Mean*	*SD*	*Comparison of sig.*	
					Tukey's HSD	*P.value*
G1 Tear strength before aging	**Control (a)**	10	19.35	1.76	a vs b	0.028 S
	$CaCo_3$ (0.5%) (b)	10	21.41	1.54	a vs c	< 0.01 HS
	$CaCo_3$ (0.75) ©	10	22.62	1.49	a vs d	< 0.01 HS
	$CaCo_3$ (1%) (d)	10	25.15	1.42	b vs c	0.32 NS
					c vs d	< 0.01 HS
G2 Tear strength after aging	**Control (a)**	10	16.73	1.58	a vs b	0.73 NS
	$CaCo_3$ (0.5%)(b)	10	17.18	0.86	a vs c	< 0.01 HS
	$CaCo_3$ (0.75) (C)	10	18.29	0.54	a vs d	< 0.01 HS
	$CaCo_3$ (1%) (d)	10	19.02	0.61	b vs c	0.07 NS
					c vs d	0.37 NS

Table (3): Comparison of the Tear strength test before and after aging by groups using a Paired Sample t-test

Studied groups	*Group A*		*Group B*		*t*	*df*	*P value*	*ES*
	Mean	*SD*	*Mean*	*SD*				
Control	19.35	1.76	16.73	1.58	4.583	9	< 0.001 HS	0.736
$CaCo_3$ (0.5%)	21.41	1.54	17.18	0.86	8.745	9	< 0.001 HS	0.871
$CaCo_3$ (0.75)	22.62	1.49	18.29	0.54	11.018	9	< 0.001 HS	0.896
$CaCo_3$ (1%)	25.15	1.42	19.02	0.61	14.783	9	< 0.001 HS	0.947

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