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Empirical study of alginate impression materials by customized proportioning system

Karani Kurtulus*, Kenan Tüfekci
Suleyman Demirel Universitesi Muhendislik Fakultesi - Mechanical Engineering Dept. Isparta, Turkey

PURPOSE. Alginate mixers available in the market do not have the automatic proportioning unit. In this study, an automatic proportioning unit for the alginate mixer and controller software were designed and produced for a new automatic proportioning unit. With this device, it was ensured that proportioning operation could arrange weight-based alginate impression materials. **MATERIALS AND METHODS.** The variation of coefficient in the tested groups was compared with the manual proportioning. Compression tension and tear tests were conducted to determine the mechanical properties of alginate impression materials. The experimental data were statistically analyzed using one way ANOVA and Tukey test at the 0.05 level of significance. **RESULTS.** No statistically significant differences in modulus of elastisity (P>0.3), tensional/compresional strength (P>0.3), resilience (P>0.2), strain in failure (P>0.4), and tear energy (P>0.7) of alginate impression materials were seen. However, a decrease in the standard deviation of tested groups was observed when the customized machine was used. To verify the efficiency of the system, powder and powder/water mixing were weighed and significant decrease was observed. **CONCLUSION.** It was possible to obtain more mechanically stable alginate impression materials by using the custom-made proportioning unit. ***[J Adv Prosthodont 2016;8:372-9]***

KEYWORDS: Alginate impression material; Alginate mixer; Mechanical properties of alginate; Preparing methods of alginate

INTRODUCTION

Irreversible hydrocolloid materials are commonly used for both diagnostic and definitive impression procedures.[1] Impression yields a detailed recording of the mouth plaster model. To obtain a mouth plaster model, impression material is used for a negative imprint of tissues in mouth. Without an impression model, it is impossible to make prosthesis. Due to their low cost and ease of use, alginate impression materials have been widely used for the past sixty years in dentistry.[2,3] Alginate mixing can be done in three different ways: manual, semi-automatic, and full automatic. Mixing by a machine decreases air entrapment and viscosity and thereby results in a better alginate paste.[4]

While alginate impression materials indicate high mechanical properties to ensure their stability during dental treatments,[5] alginate impression materials are required to have enough viscosity to make a way into all oral structures. There are various mechanical tests applied to impression materials and their mechanical properties, such as ANSI/ADA specification no. 18-1992[6] and ISO 37:2005 specifications. Based on these specifications, the mechanical properties, such as elastic recovery, strain under compression, tensile strength, compression strength, and tear energy, are considered.[7-9]

Recent studies showed that performing automatic alginate mixing showed various effects on alginate materials - commercially named Identic (Cadco), Kromopan (Lascod), and Jeltrate (Dentsply/Caulk).[4,10] While an increase in the tear energy was observed in the Identic alginate, it was not observed in the other alginate materials. The three alginates showed higher elastic recovery and compressive strength for automatic mixing when compared to manual mixing.

Although alginate manufacturers represented the powder/water ratio as the weight ratio in the past, volume-

Corresponding author:
Karani Kurtulus
Suleyman Demirel University Mechanical Engineering Dept.
Sünür, Isparta, 32260, Turkey
Tel. +905422787499: e-mail, karanikurtulus@sdu.edu.tr
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based proportioning has been preferred because it is much easier in the practice. 10 - 20% weight difference is acceptable for use.[11] In this study, an automatic proportioning unit with weight-based proportioning for the alginate mixer machine was designed. It was then assembled to the mechanical mixer. Mechanical properties were tested for the alginate impression material obtained by using the customized weight-based proportioned-automatically mixed (WP-AM) machine. Mechanical properties of the alginate impression materials by WP-AM machine was statistically compared to those by volumetrically proportioned-manually mixed (VP-MM) and volumetrically proportioned-automatically mixed (VP-AM).

MATERIALS AND METHODS

While designing the proportioning unit for the alginate mixer machine, criteria, such as ergonomics, simple structure, and low cost, were considered with the dentists' advice. The working principle of the device and the dust hopper of the designed alginate proportioning unit are shown in the Fig. 1, respectively.

The density of the alginate powder is 0.452 g/cm^3 and the particle size range is between 20 - 50 µm. It consists of various chemical substances. Due to its being such a fine powder mixture, problems such as powder sticking to the walls of the dust hopper and other components (blades and screw conveyor, etc.) may occur. In order to minimize these problems, the gravity was taken into consideration by placing a screw conveyor vertically (Fig. 1B).

There were PIC18F452 microprocessor, RS 232 communication port, L298 motor driver, 74C922 keypad integrity, and display on the electronic control card specially designed for the device. The desired powder/water ratio for different alginate material was recorded to the system by using the keypad. Desired amount of alginate powder was transferred to the alginate container by rotating the screw conveyor with electronically controlled motors. Alginate container was placed on the load cell of the electronic scale with 0.1 g sensitivity. The microprocessor constantly controlled the weight of the container and stopped the powder flow by terminating the rotation movement of the screw conveyor when the weight of the powder had reached its desired value. Then, the electromagnetic valve began the water proportioning operation. When the water taken from the tank reached the desired level, the electronic valve was closed and the user was informed with "Proportioning operation is completed!" signal message. During all these operations, net weight of the powder/water in the container could be monitored on the screen. The assembled form of the machine and its cross-sectional view are shown in Fig. 2. As seen in Fig. 2, the main device included (1) mixing unit, (2) dust hopper, (3) scale module, (4) power supply, (5) electronic control card, (6) load-cell, (7) propulsion motors, (8) basement, and (9) dust hopper holders.

The program controlling the system was written on C++, and later it was embedded in the microprocessor with PIC programming circuit. Various protection measures were taken by the software because it was possible that powder/water ratios were entered wrong by the dentist. In such case, it might cause damage to the device as a result of the overflow of the alginate container. Powder/water weight ratio suggested by the manufacturer can be adjusted at a definite ratio by multiplying with a coefficient called "filling coefficient"; thus, material amount can be controlled. For example, when the filling coefficient with the default value of 1.0 is entered as 1.1, powder and water weights registered in the system can be increased at the rate of 10%. Similarly, it is decreased at the rate of 10% when it is entered as 0.9.

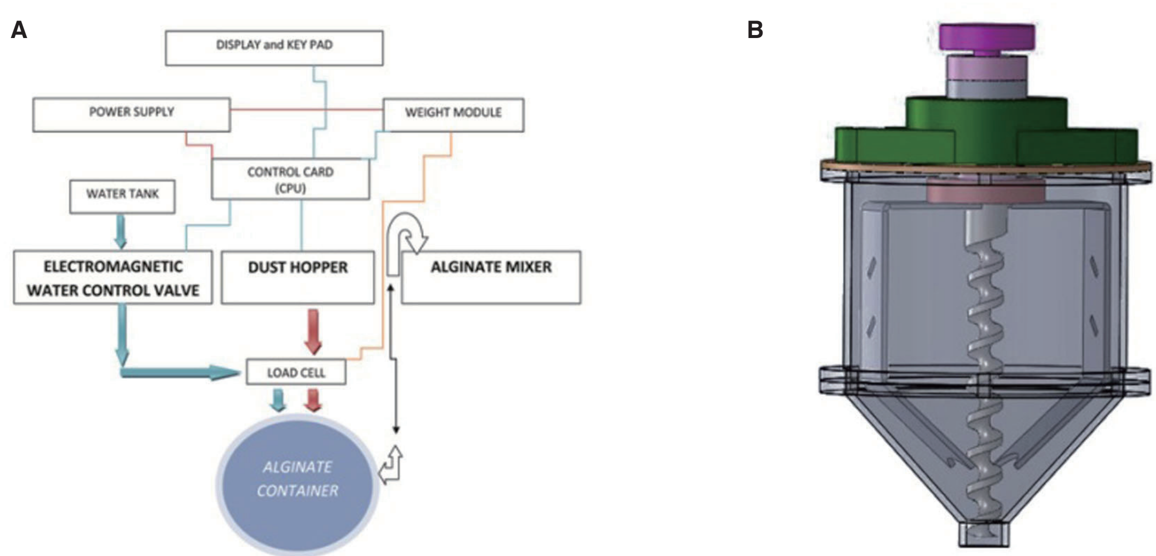

Fig. 1. (A) Working principle of the machine and, (B) Cross-section view of the dust hopper.

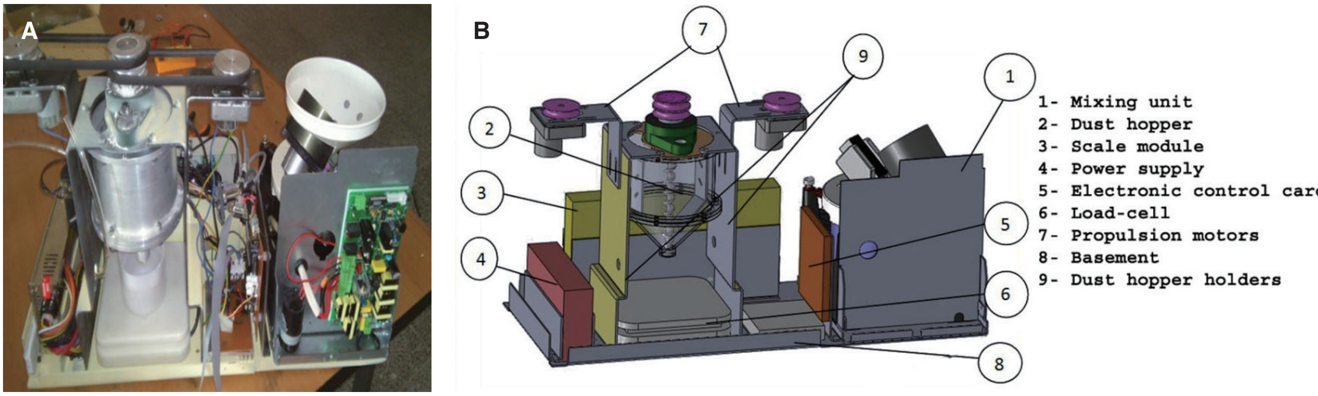

Fig. 2. A photograph of the fully assembled device (A) and its schematic illustration (B).

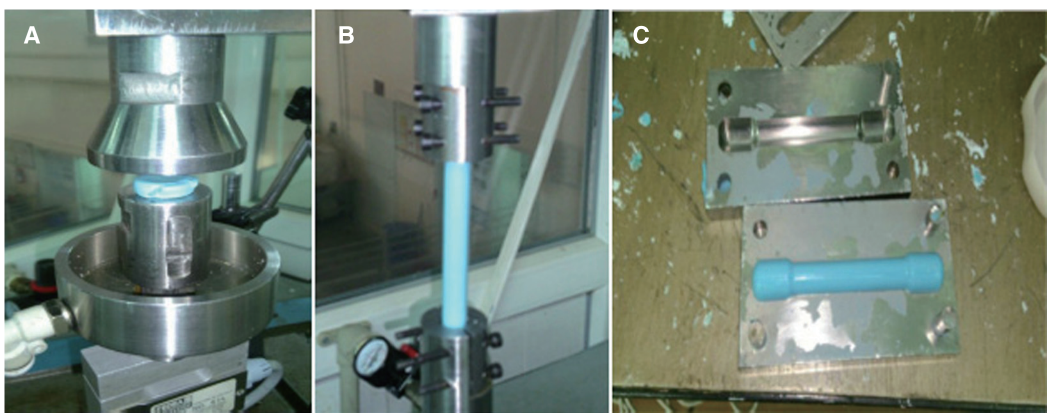

Fig. 3. (A) Compression test setup, (B) Tensile test setup, (C) Split mould for tensile test specimen.

Specimens for tensile, compression, and tearing tests were prepared by three different methods. Alginate impression material was manually proportioned (volumetrically) and manually mixed (VP-MM) in Method I, manually proportioned and automatically mixed (VP-AM) by the machine in Method II, and the proportioning operation was carried out with the machine (weight-based) and automatically mixed by the machine (WP-AM) in Method III. VP-MM and VP-AM are commonly used in clinical settings. They were tested by preparing 8 specimens for each group, summing up to seventy-two specimens in total. The specimens were prepared by using Cavex Impressional as an alginate impression material. All manual mixing processes were conducted according to manufacturer's directions by the same dentist who had five years of experience.

Compression (Fig. 3A), tensile (Fig. 3B), and tear tests for WP-AM method were compared to the mechanical properties of the specimens obtained with the VP-MM and VP-AM methods.

The alginate impression material prepared by VP-MM method was proportioned into a tray. Measured water (by cup) and alginate impression material were manually mixed for 45 seconds with a spoon. The specimens prepared with the VP-AM method were proportioned in the same way as the VP-MM method and were mixed for 10 seconds by a mechanical mixer. In the specimens prepared with the WP-AM method, proportioning and mixing were carried out automatically. The upper and the lower plates of the split mould were kept assembled for the whole setting time, as recommended by the manufacturer. Then, the mould was opened, and the test specimens were removed from the mould. Re *et al.*[5] indicated that there were significant effects of the waiting period on both viscosity and mechanical properties of the alginate impression materials. Therefore, all tests were conducted after enough setting times.

Compression tests were carried out by following ANSI/ADA Specification No. 18-1992 Standards.[6] For tensile and compression tests, specimens were obtained by using the cylindrical dumbbell specimens of the same diameter. In order to prepare the specimens of the dumbbell shape, the split mould for the tensile test specimen, as shown in Fig. 3C, was designed by using CAD (computer aided design) system and manufactured on a CNC milling machine.

A custom-made testing machine was specially designed at the Department of Mechanical Engineering, Suleyman Demirel University (SDU) in Isparta, Turkey, in order to conduct the mechanical tests of the materials with low strength. By using this machine, static tension and compression tests were carried out under displacement control at an average rate of 0.5 mm/s. In order to measure the load and corresponding displacement while conducting tests, the custom-made testing machine was equipped with a load cell of 50 kg (Tedea Huntleigh MN:16, Malvern, PA, USA) and two LVDTs (Novotechnik, Ostfildern, Germany).

The LVDTs were fixed to the lower loading platen using a holder and positioned near the upper loading platen at a nearest location to the sample using a custom-made fixture for deformation measurements. The readings got from the two LVDTs were averaged, and the average deformation was calculated for each specimen. Afterwards, the calculated average deformation was converted into strain by using the thickness of the specimen. In order to prevent an overestimation of the strain, the deformations in the load cell, in the test rig, and in the overall machine structure were eliminated as the axial strain was calculated with the displacement of the upper loading platen relative to the lower platen at the nearest point to the sample (Fig. 4). A four-channel oscilloscope (Nicholet-Oddysey XE, Madison, WI, USA) was used in order to take records of the data measured from both transducers at the rate of 100 data points per second.

A stress-strain curve of the selected VP-AM sample, which was obtained from the tensile and compression tests, is shown in Fig. 5. It was observed that along with increasing tensile load, stress in the material increased until it reached its maximum value. When loading was continued, necking occurred at a certain point (usually in the area close to the middle point) of the material. In this point, the cross-sectional area of the material decreased rapidly, the load that it could carry decreased, and later damage occurred. It was easy to find the failure point on the tensile test curve. As it is seen in Fig. 5, the point at which the force data began to become sparse was the failure point. In the compression test, the material showed similar characteristics. However, it was harder to detect the failure point during the test. Even after the damage occurred in the material, the damaged specimens still continued to carry the load. For this reason, a sudden dilution or break in the force data was not observed as in the case of the tensile test. In this study, camera images and force-extension data were analysed to detect the damage point in compression tests. It was observed that the force remained fixed soon after the damage had happened. Moreover, at the determined failure point for the compression specimen, the force reached its peak value, decreased slightly, and began to move horizontally (Fig. 5).

Stress (σ) and strain (ε) values were calculated with the equation (1) and equation (2); where, F: force, s_0: initial cross-section area, l_0: initial length, and l_f: final length value, respectively. The material's modulus of elasticity value was calculated from the slope of the linear portion of the compression-tensile curves. The ultimate stress value was the highest stress value of the stress-strain curve. The resilience was calculated to be the area under the linear part of the stress-stain curve. Resilience was defined as the maximum energy that can be absorbed within the elastic limit without creating a permanent deformation. This value was especially high in the tensile test, which was important for the alginate impression material to keep its original shape and size during the removal from the mouth.

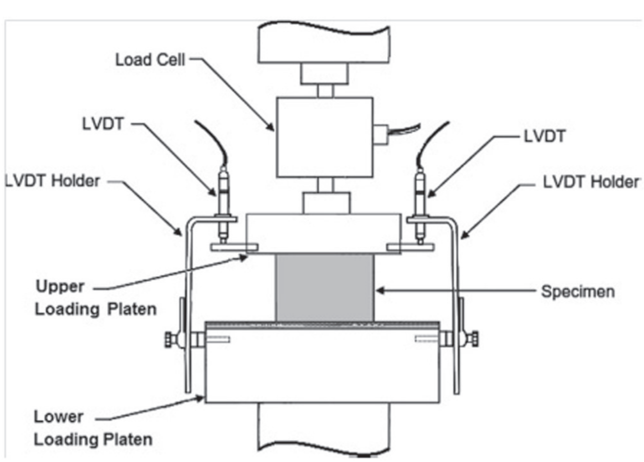

Fig. 4. A schematic diagram of the LVDTs installation for compression test.[12]

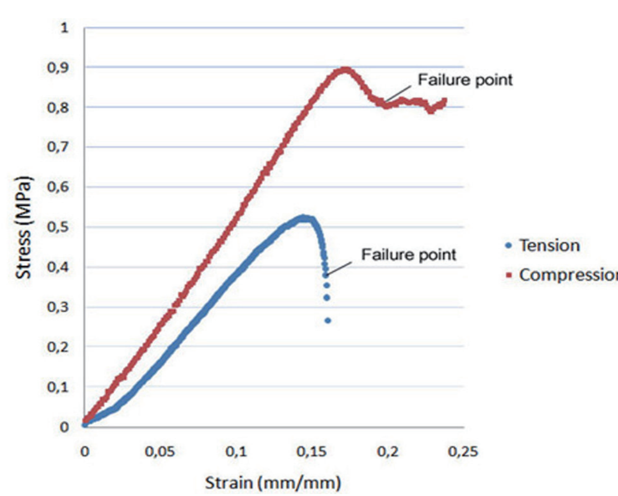

Fig. 5. Stress-strain curves of the selected VP-AM sample.

$$\sigma = \frac{F}{s_0} \quad (1)$$

$$\varepsilon = \frac{l_f - l_0}{l_0} \quad (2)$$

While there are many studies on tear resistance in the literature,[13-15] there has not been any standardized method mentioned yet. For this reason, it is hard to evaluate the data obtained from different studies. While ANSI/ADA standards require the tear resistance of alginate impression materials to be tested an hour after polymerization, impression materials are exposed to tensile forces right after the setting time in the clinical applications. In this study, tear energy tests were carried out right after the setting time compatible with the clinical applications by using trouser leg-shaped specimens as suggested by Webber and Ryge.[16]

RESULTS AND DISCUSSIONS

The weight of both the powder and powder/water obtained after manual proportioning and proportioning with the machine were measured by an electronic scale with 0.001 g sensitivity. The coefficient variations of volume- and weight-based proportioning of powder and powder + water mixing are shown in Table 1. Caswell stated that the alginate between powder and powder + water mixture volumetrically proportioning deviated between 10 - 20% in contrast to weight-based proportioning. This ratio was found to be around 7.5% in this study. The coefficient of variation remained around 1% when the proportioning was done by the mechanical mixer.

The means and standard deviations of the mechanical properties obtained from the compression experiments are shown in Fig. 6. According to ANSI/ADA Specification No. 18,[17] the minimum compression strength must be around 0.35 MPa. The values of the compression strength obtained from all three methods were higher than the standard values. The failure strain obtained from all the methods was similar to the limits as well (5 - 20%).

Table 1. Coefficient of variation (CV) of powder and powder + water mixing weight

	CV	
	Volumetric proportioning	Weight-based Proportioning
Powder (6 g)	7.7%	1.1%
Powder (6 g) + Water (15 g)	7.3%	0.9%

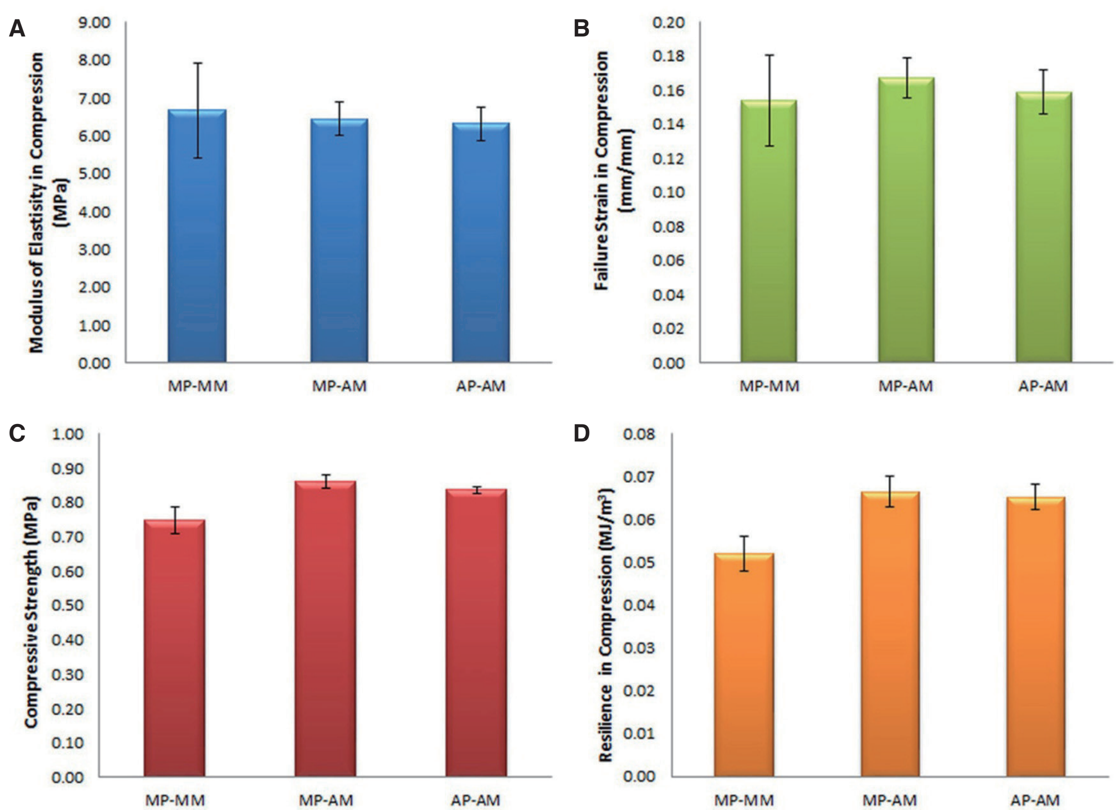

Fig. 6. Mechanical properties of VP-MM, VP-AM, and WP-AM under compression. (A) Modulus of elastisity, (B) Failure Strain, (C) Compressive Strength, (D) Resilience.

Lee *et al.*[18] focused on the effect of adding fluoride on the mechanical properties of the dental alginate impression material. In the compression tests they carried out in accordance with ISO 1563 standards, the modulus of elasticity values of the control group specimens were found to be 4.3 MPa, and the compression strength values were found to be around 1.2 MPa. However, Frey *et al.*[10] found that the compressive strength value to be between 0.7 - 0.9 MPa and strain in failure value to be between 13 - 15% from the compression tests with three different alginate impression materials under the same standards. The values found in this study were compatible with the values in both studies.

Nallamuthu *et al.*[19] calculated the elasticity modulus of some commercial alginate impression materials (Neocolloid, Pulgat Plus, Blue Print Cremix) using shore hardness test which is an indirect method suggested by Gent[20], and they found this value to be between 0.7 - 0.9 MPa. The values calculated using indirect methods were lower than the modulus of elasticity values obtained in our study and Lee's[18] study.

The means and standard deviations of the mechanical properties obtained from the tensile test are shown in Figure 7. While the failure strain value obtained from the tensile and compression tests demonstrated the values close to each other, other mechanical properties were found to be higher in the compression test. This case has important implications in terms of clinical applications. The resilience value turned out to be low in the tensile test, meaning that the alginate impression material would be deformed much more easily under the tensile stress. So, alginate impression material is torn easily when subjected to tensile stress during the removal operation from the mouth. Increasing the resilience value (with additives or with different processes) in the tensile test would be important for the researchers who work on the improvement of the mechanic properties of alginate impression materials.

The mechanical properties obtained from tear energy tests are shown in Fig. 8. Frey *et al.*[10] figured out that there were statistically significant differences between the tear energies of different types of alginates. In the same study with the alginate impression materials of Jelrate and Kromopan, while no effect of the preparation method (hand mixing, manual mixing) was seen on the tear energy value, a statistically significant difference was found in the Identic. Nallamuthu *et al.*[19] also found higher tearing energy than our study and Frey *et al.*'s[10] study for the commercially alginate materials of Neocolloid and Palgat Plus as 303, 323 J/m^2.

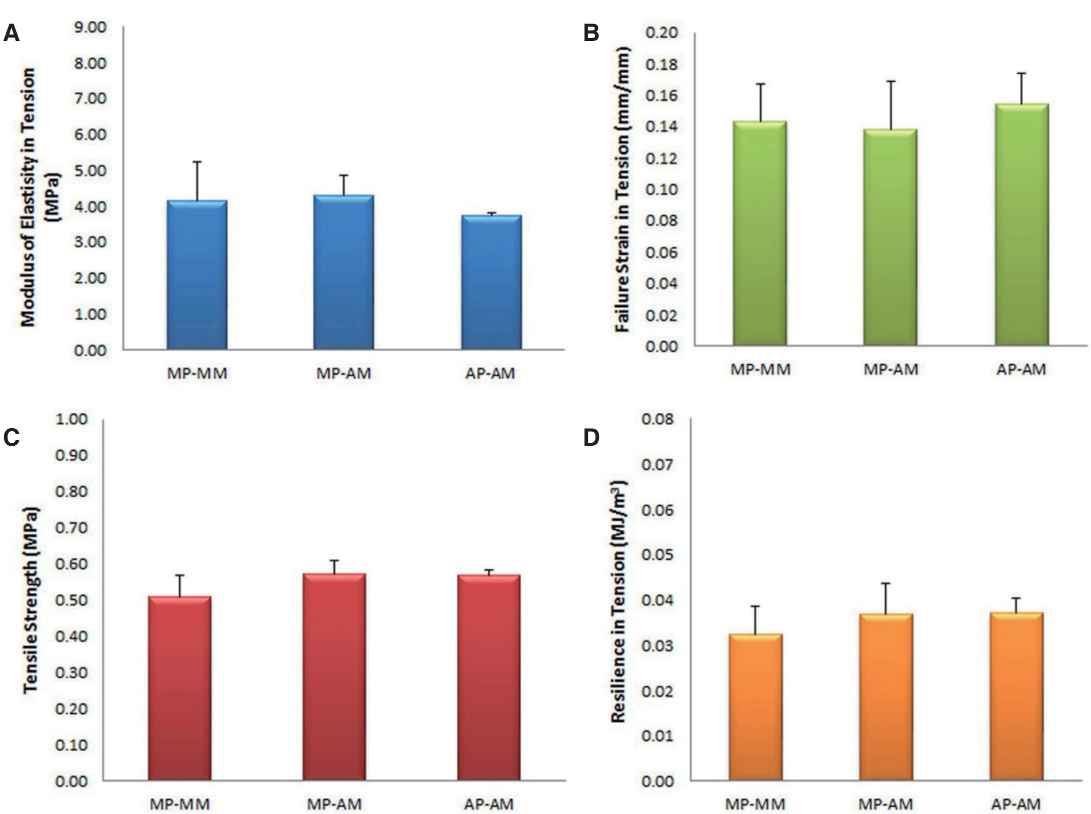

Fig. 7. Mechanical properties of VP-MM, VP-AM, and WP-AM under tension.(A) Modulus of elasticity, (B) Failure Strain, (C) Compressive Strength, (D) Resilience.

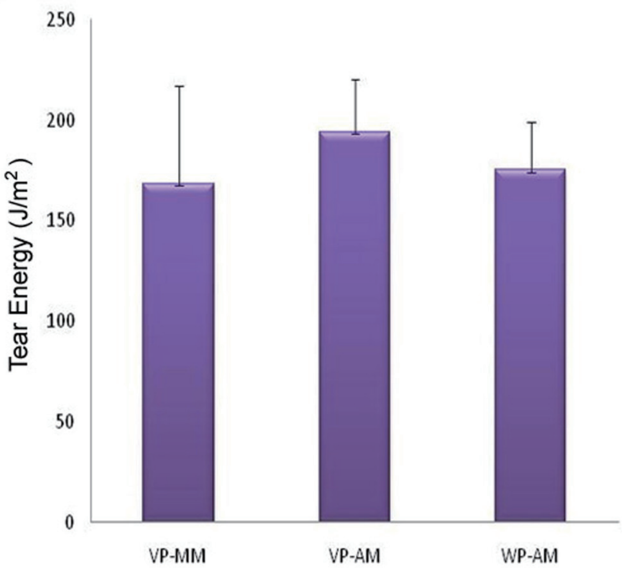

Fig. 8. Tear energy values of VP-MM, VP-AM, and WP-AM.

The results of the one-way variance analysis for the alginate prepared with three different methods are shown in Table 2. While significant differences were observed between groups in the compression strength and resilience values in the compression test, no significant differences in the strain at failure and modulus of elasticity values were observed. Furthermore, significant differences were observed in the tensile strength. No significant difference was found between the groups in tear energies. Tukey's comparison test showed that the test difference originated from Method 1 (VP-MM). Statistically significant differences were not observed between Method II (VP-AM) and Method III (WP-AM).

These results showed that manual (hand) mixing or machine mixing had a greater effect on mechanical properties of the alginate impression material. Inoue[4] indicated that carrying out the mixing operation with a machine decreased bubbles in the material. During the machine mixing, alginate paste and air bubbles are exposed to a very high centripetal acceleration. The bubbles are separated from alginate paste under a high centripetal acceleration since the density of bubbles is much lower than density of the alginate paste. Thus fewer bubbles remain within the alginate paste.

When the proportioning operation was carried out with the customized machine, although no statistically significance differences in the mechanical properties of alginate impression materials were seen, a decrease in the standard deviation was observed. Therefore, all alginate paste specimens would have similar mechanical properties and this is desired for the clinical applications. Installing the automatic proportioning unit to alginate mixers would increase the volume that the device occupies by 80%. It would also increase the cost by 50% (it might change according to the production method and technology). A limitation of this study was that only one alginate material was investigated. Future study should follow using different alginate materials to show different mechanical behaviour when the proportioning operation is conducted on the customized machine used in this research.

CONCLUSION

In case the proportioning operation is carried out volumetrically, while the deviation from the weight suggested by the manufacturer was 7.5%, the deviation remained to be around 1% when the device was used. The significant increases in the compressive strength, tensile strength, and resilience in compression values were observed when the mixing operation of the alginate impression material was carried out with the customized machine. While no increase in the mechanical properties was observed when the proportioning operation was carried out automatically, a decrease in the standard deviation values was observed. Therefore, using the customized device with the automatic proportioning unit in the alginate mixing operation will ensure obtaining more stable specimens. It will also shorten the time spent for paste preparing and prevent material waste.

Table 2. One-way variance analysis for the alginate prepared with three different methods (VP-MM, VP-AM, WP-AM)

Mechanical Properties	Compression		Tension		Tear Energy	
	F	*P*	F	*P*	F	*P*
Modulus of Elasticity	0.24	.79	1.31	.29	0.31	.74
Comp./Tens. Strength	31.41	< .001	5.18	.02		
Resilience	24.88	< .001	1.76	.2		
Strain at Failure	0.66	.53	0.97	.4		

NOMENCLATURE

Symbols	
ε	strain (mm/mm)
µm	Micro meter
g	gram
F	force (N)
l_0	initial length (mm)
l_f	final length (mm)
σ	Stress (MPa)
J	Joule

Acronyms	
ADA	American Dental Association
ANSI	American National Standart Institue
ISO	international Organization of Standardization
WP-AM	weight-based proportioned-automatically mixed
VP-MM	volumetrically proportioned-manually mixed
VP-AM	volumetrically proportioned-automatically mixed
CAD	computer aided design
LVDT	linear variable differential transformer

ORCID

Karani Kurtulus *http://orcid.org/0000-0002-2608-3065*
Kenan Tüfekci *http://orcid.org/0000-0002-3036-6375*

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