Development of Elastic Ligatures from Natural Rubber

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บทคัดย่อ

ยางที่ผลิตจากยางสังเคราะห์เป็นที่นิยมใช้ในงานทันตกรรมจัดฟันอย่างกว้างขวาง แต่พบว่ายางที่ผลิตจากยางสังเคราะห์จะมีการสูญเสียแรงมากเมื่อเวลาผ่านไปเมื่อเทียบกับยางธรรมชาติ ขณะที่ยางธรรมชาติมีคุณสมบัติการสูญเสียแรงต่ำกว่าจึงเป็นเรื่องน่าสนใจที่จะพัฒนาผลิตยางยึดฟันจากยางธรรมชาติ **วัตถุประสงค์** เพื่อผลิตยางยึดฟันจากยางธรรมชาติ (LE) และศึกษาเปรียบเทียบเปอร์เซ็นต์การสูญเสียแรงและการสูญเสียรูปร่างอย่างถาวรระหว่างยางยึดฟันที่ผลิตจากยางสังเคราะห์ (UNI) กับยางยึดฟันที่ผลิตจากยางธรรมชาติ **วัสดุและวิธีการ** ผลิตยางยึดฟันจากยางธรรมชาติและทำการเปรียบเทียบคุณสมบัติกับยางยึดฟันจากยางสังเคราะห์จากบริษัท Unitek (Alastik A1 module) ตัวอย่าง 60 ตัวจากแต่ละชนิดมาแบ่งออกเป็น 2 กลุ่มกลุ่มละ 30 ตัว ตัวอย่างทุกตัวถูกยึดบนแท่นเหล็กกล้าไร้สนิมเส้นรอบวงเท่ากับแบรตเกตขนาดใหญ่พร้อมกับลวดและแช่ในน้ำลายเทียมที่อุณหภูมิ 37 $^{\circ}$ C กลุ่มแรกทำการศึกษาถึงการสูญเสียแรง โดยการวัดขนาดของแรงที่ระยะ 5.5 มิลลิเมตร ที่เวลาเริ่มต้น 1 วัน 7 วัน 14 วัน และ 28 วัน กลุ่มที่สองทำการศึกษาเกี่ยวกับการเปลี่ยนแปลงรูปร่าง โดยที่การวัดขนาดเส้นผ่าศูนย์กลางภายใน เส้นผ่าศูนย์กลางภายนอก และความหนาของผนังของยางยึดฟัน ที่เวลาเริ่มต้นและ 28 วัน วิเคราะห์ทางสถิติด้วย Repeated measures ANOVA เพื่อทดสอบความแตกต่างอย่างมีนัยสำคัญระหว่างเปอร์เซ็นต์การสูญเสียแรงในยางยึดฟันแต่ละชนิดและแต่ละช่วงเวลา independent pair *t* test เพื่อทดสอบความแตกต่างอย่างมีนัยสำคัญระหว่างเปอร์เซ็นต์การสูญเสียแรงและเปอร์เซ็นต์การเปลี่ยนแปลงรูปร่างระหว่างยางยึดฟัน 2 ชนิดที่ช่วงเวลาเดียวกัน dependent pair *t* test เพื่อทดสอบความแตกต่างอย่างมีนัยสำคัญระหว่างรูปร่างเริ่มต้นกับรูปร่างสุดท้ายที่ 28 วันในยางยึดฟันแต่ละชนิด และหาความสัมพันธ์ระหว่างเปอร์เซ็นต์การสูญเสียแรงกับขนาดของแรงเริ่มต้นและเปอร์เซ็นต์การเปลี่ยนแปลงรูปร่างระหว่างทำการศึกษา **ผลการทดลอง** (1.) LE มีเปอร์เซ็นต์การสูญเสียแรงที่ต่ำกว่า (28.26 %) เมื่อเทียบกับ UNI (72.31 %) ที่ระยะเวลา 28วัน (2.) เปอร์เซ็นต์การสูญเสียแรงจะสูงที่สุดในช่วง 1 วันแรก (16 % ใน LE และ 66 % ใน UNI) (3) LE มีเปอร์เซ็นต์การเปลี่ยนแปลงรูปร่างต่ำกว่า UNI โดยเฉพาะในส่วนของเส้นผ่าศูนย์กลางภายใน (24.34% สำหรับ LE ขณะที่ 191.30% สำหรับ UNI).

สรุป จากการศึกษานี้แสดงให้เห็นว่ายางยึดฟันที่ผลิตจากยางธรรมชาติเหมาะสมสำหรับเคลื่อนฟัน เนื่องจากมีการสูญเสียแรงและการเปลี่ยนแปลงรูปร่างที่น้อย

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ABSTRACT

Elastomeric products are widely use in orthodontics. Unfortunately, the forces of elastomeric degrade overtime more than force of latex elastic. Therefore it is interesting to produce elastic ligatures from natural rubber. **Objectives**: To develop latex elastic ligatures (LE) with low force degradation and small permanent deformation at Prince of Songkla University (PSU), Thailand and to compare the percentages of force degradation and the percentages of permanent deformation between LE and clear non-latex elastic ligatures (Unitek ®, UNI) in 37 ° C artificial saliva. **Materials and methods**: Natural rubber was used to produce the LE to compare to the UNI. All tested groups (30 ligatures for each group) were stretched over stainless steel dowels with a circumference approximating that of a large orthodontic twin bracket with arch wire in a synthetic saliva bath at 37 ° C. The force level was measured using the universal testing machine stretching for 5.5 mm. at initial, 24 hours, 7 days, 14 days, and 28 days respectively. For the permanent deformation test, initial wall thickness, inside diameter, and outside diameter were measured at initial and 28 days respectively. Means and standard deviations were calculated. Repeated ANOVA was used to compare the means of percentage of force changes. Independent pair *t* test was used to compare the percentage of dimensional changes and the percentage of force degradation between LE and UNI in the same time. Dependent pair *t* test was used to compare the initial dimensional and the final dimensional between LE and UNI. Correlation coefficients were calculated to determine the relationship between the percentage of force degradation and initial force level and the percentage of permanent deformation at the initial and after 28 days stretching period. **Results**: The results for stretched samples in a simulated oral environment revealed the followings: (1) LE has little gradually force loss (28.26 %) compared to UNI (72.31 %) from initial to 4 weeks. (2) The greatest force loss is statistically significant occurred in the first day (16.30 % in LE and 66.07 % in UNI), (3) LE has statistically significant lower percentage of permanent deformation than that of UNI especially for the inner diameter

(24.34 % for LE while 191.30 % for UNI). **Conclusion**: This reveals that LE made at PSU is suitable for moving tooth due to its less percentage of force deformation and permanent deformation.

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Finally, I would like to give my special thanks to my parent, dad and mom, and everyone in my family for their encouragement and support throughout my study.

Chainarong Kaosampan

CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS AND SYMBOLS

ANOVA	=	One-way analysis of variance
º C	=	Degree Celsius
cm	=	Centimeter
Fig.	=	Figure
FDA	=	Food and Drugs Administration of the United States of America
g	=	Gram
hr	=	Hour, hours
ie	=	Immunoelectrophoresis
ID	=	Inside diameter
kN	=	Kilo newton
kPa	=	Kilo Pascal
LE	=	Latex elastic ligatures
mm	=	Millimeter
min	=	Minute
MPa	=	Mega Pascal
OD	=	Outside diameter
pH	=	Potential of hydrogen
phr	=	Per hundred
s	=	Second
SD	=	Standard deviation
UNI	=	Unitek ® non-latex elastic ligatures
WT	=	Wall thickness
®	=	Trade name
/	=	Per
%	=	Percentage

CHAPTER 1

INTRODUCTION

1.1 Background and rationale

Elastomer is a general term that encompasses materials that, after substantial deformation, rapidly return to their original dimensions. Natural rubber, probably had used by the ancient Incan and Mayan civilizations, was the first known elastomer. It had limited use because of its unfavorable temperature behavior and water absorption properties. With the advent of vulcanization by Charles Goodyear in 1839, uses for rubber greatly increased. Early advocates of natural rubber latex elastics in orthodontics included Baker, Case, and Angle[1]. Due to possible allergic natural rubber latex protein, synthetic rubber polymers made of polyurethane was introduced in the 1920[2].

Orthodontists may use 0.008 to 0.014 inch stainless steel ligature wire, self-ligating spring clips, or circular synthetic elastomers to secure arch wires to orthodontic brackets. Advertised characteristics of elastomeric ligatures include: continuous gentle forces, consistent long-lasting arch wire seating, water sorption resistance, and shape memory properties. The elastomeric ligatures regularly made of polyurethanes which exact composition is a commercial secret. Orthodontists use elastomeric ligatures for engaging the arch wire to a bracket slot, closing space and correcting rotation. The advantages of elastomeric ligatures are that they can be applied quickly, patient – friendly nature, aesthetic appearance and potential for fluoride release. Disadvantages are rapidly force degradation as well as permanent deformation, arch wires may not completely seat during torquing or rotational corrections, and binding may occur during sliding mechanics[3].

At the leveling phase, elastomeric ligatures can be either active to pull the brackets toward arch wires or passive by strongly holding the brackets with the arch wires then let the arch wires to move the brackets. After leveling, arch wires need to be firmly seated in the bracket slots so strong elastomeric ligatures are recommended. According to their purposes, elastomeric ligatures should be made to have either high elasticity for moving teeth or low elasticity for holding the bracket with the wire.

Natural rubber has been used for making many products in Thailand. High elasticity products as rubber bands and glove or low elasticity products as automobile type could be made in Thailand. This available technology is possible to apply for producing elastomeric ligatures with either high or low elasticity.

1.2 Review of the literature

The elastomeric ligatures, in general, are polyurethanes, which are thermosetting polymers possessing a -(NH)-(C=O)-O- structural unit and formed by step reaction (condensation) polymerization. The elastomeric properties of these materials are derived from the phase separation of the hard and soft copolymer segments of the polymer, such that the urethane hard segment domains serve as cross-links between the amorphous polyether (or polyester) soft segment domains. The hard segments are considered held together in discrete domains through the action of Van der Waal's forces and hydrogen bonded interactions. The soft segments, which are formed from high molecular weight polyols, are mobile and are normally present in coiled formation, while the hard segments, which are formed from the isocyanate and chain extenders, are stiff and immobile. Because the hard segments are covalently coupled to the soft segments, they inhibit plastic flow of the polymer chains, thus creating elastomeric resiliency. Upon mechanical deformation, a portion of the soft segments are stressed by uncoiling, and the hard segments become aligned in the stress direction. This reorientation of the hard segments and consequent powerful hydrogen bonding contributes to high tensile strength, elongation, and tear resistance values.[4, 5] Elastomeric ligatures are manufactured in two basic forms: injection molded and cut. The injection molded ligature is made by injection of liquefied elastomeric material into a mold and curing, whereas the cut ligature is sliced from previously processed elastomeric tubes. In addition, the polymer structure of the polyurethane modules may differ among companies.[3] These products are not ideal elastics and are affected by duration of force and environment.

Elastomeric ligatures are regularly used for exerting force on arch wires and for space closure and for rotational corrections. Mclaughlin, Bennett and Trevisi[6-8] suggested that during sliding mechanics for closing the space, light continuous forces are applies using elastic tiebacks (single non latex elastic modules to anterior arch wire hooks with ligature wires extended forward from the molars, After 2-3 mm stretching, the modules generate about 100-150 g of force). Non latex elastic modules stretched by 2-3 mm usually close 0.5-1.5 mm of space for a mouth. The tiebacks are replaced every four to six weeks.

A study of the efficiency of space closure after premolar extraction by Samuels[9] was undertaken, comparing a nickel-titanium closed coil spring and a non latex elastic retraction module (The module was activated by 2 to 3 mm stretching, or twice the diameter of the module in every patient providing a starting force of 400 to 450 g as measured clinically by a strain gauge) by using sliding mechanics along an 0.019 × 0.025 inch stainless steel arch wire in 0.022 × 0.028 inch pre-adjusted stainless steel brackets. The rate of space closure in 17 subjects was analyzed from study models and was found to be significantly greater and more consistent with the nickel-titanium closed coil springs than with the non latex elastic modules, in both arches. There were no clinically observable differences in the tooth positions between the respective techniques but at the subsequent visit (6 weeks), the force provided by the spring was unchanged, whereas that of the elastic module had reduced to approximately zero. Therefore, nickel-titanium closed coil springs may be able to achieve rapid closure due to their low constant force, whereas with elastic modules, the closure rate might relate to the intermittent force.

Abrahamian[10] suggested that elastomeric ligatures can be used to correct individual tooth malposition with fixed appliances. To rotate a tooth distolingually, tie elastomeric ligatures in a figture-8 to the distal wing of the bracket. After placing the archwire, tie the mesial wing of the bracket to the archwire with a ligature wire or an elastic tie.

The force degradation rate, the amount of force degradation within a specific time, the dimensional changes of elastomeric ligatures related to the force degradation has been reported. Huget et al[11] evaluated and focused on the changes in elasticity of a synthetic orthodontic elastomeric (polyurethane) storage in water. The experiment in water was indicated that exposure of the elastomeric to water leads first to weakening of non-covalent forces and subsequently to degradation.

Taloumis et al[12] evaluated commercially available molded gray elastomeric ligatures from seven companies for the force degradation, dimensional change, and the relationship between ligature dimension and force. The results for stretched samples in a simulated oral environment revealed the followings: (1) moisture and heat had a pronounced effect on force degradation and permanent deformation, (2) a positive correlation existed between the wall thickness and force, (3) a negative correlation existed between the inside diameter and force, (4) a weak correlation existed between outside diameter and force, (5) the greatest force loss occurred in the first 24 hours and the degradation pattern was similar for all ligatures tested, and (6) unstretched ligatures absorbed moisture in the range of 0.060% to 3.15%. The mean percentage of force degradation for 24 hours was 53% to 68% for the seven companies and was comparable to those reported in the published studies for elastomeric chains.

Thailand is the world's number one producer of natural rubber (NR). In the year 2002, it produced 2.63 million tons of raw rubber with 90% being exported as a raw material and the rest used to produce rubber products for exporting and use within this country. To increase the value added products of natural rubber, the Thai government realized the need to increase the potential and performance of rubber products manufactured in Thailand[13].

The molecular structure of natural rubber is composed of carbon atoms and smaller hydrogen atoms. However, its amorphous mass of coiled and kinked chains readily allows motion of its molecular chain making it extremely flexible. Rubber in its natural form is too soft to be used for any useful purposes. Therefore, its properties were improved using special processing techniques. In 1839, Charles Goodyear discovered a process for converting soft natural rubber into a harder, less flexible material which is known as the 'rubber' in our tires today. Vulcanization is a process in which sulfur, when combined with the natural compounds of rubber, cross links the molecular chains at their double bonds to restrict molecular movement, and increase hardness[14].

Recently, a number of studies compared the mechanical properties and relaxation characteristics of latex and non-latex elastics have demonstrated a vastly different time-related mechanical performance of non latex materials. In general, non-latex elastics have been shown to present more force degradation over time than latex elastics.

The study of the properties of latex and non-latex elastic bands has shown that the force degradation of latex elastic bands was less than that of non latex ones. Bishara and Andreasen[15] compared 3/16 inch, 1/4 inch, 5/16 inch and 6/16 inch latex and non-latex elastic bands at extension lengths of 22, 28, 34 and 40 mm. Non-latex elastic bands were examined in water at 37°C, and latex elastic bands in water at room temperature. The durations of the studies were 1 minute, 1 hour, 24 hours, 1 week, 2 weeks and 3 weeks. The force degradations of latex and non-latex elastic bands at 1 hour were 10% and 45.3%, respectively, and at 3 weeks 25.1% and 67.5%, respectively. The force degradation of latex elastic bands was lower than that of non-latex elastic bands. Their results corresponded with Kersey et al[16], who compared 1/4 inch latex and non-latex elastic bands from the same manufacturer. They measured the elastic bands at 4, 8 and 24 hour in water at 37°C by static testing, they found that the remain force in latex elastic bands and non-latex elastic bands was 87%, 85% and 83% and 83%, 78% and 69%, respectively. In dynamic testing, the remaining force was lower than in static testing. Russell et al[17] compared the force degradation of 1/4 inch latex and non-latex elastic bands from 2 manufacturers. The extension length was 2 and 3 times of the internal diameters of the elastic bands. They examined at 1 hour, 6 hours, 12 hours and 24 hours. The dry test was carried out at room temperature while wet test in water at 37°C. The results showed that force degradation was significant differences between the latex and the non-latex elastics and between the different manufacturers.

Andreasen and Bishara[18] compared latex elastic bands and non-latex elastic chains with respect to simulated intra-arch space closure and inter-arch force. They found that, after loading for 24 hours, non-latex elastics suffered a 74 % loss of force delivery capability, whereas latex elastic bands only lost 42%. Non-latex elastic chains are permanently deformed by approximately 50 % of their original length; comparatively, elastics are permanently deformed by 23 % of their original lengths.

Development of natural rubber latex at Prince of Songkla University, Patarapaiboolchai[19] reported the using of reinforcing fillers (Vulcanizing agent and accelerator) in compound latex for improving tensile strength and elasticity properties follow under the West German food law, and Food and Drugs Administration (FDA) of the United States of America.[20] This compound latex transformed to latex elastic products with heat sensitive dipping method. This method can control latex elastic thickness with both heat, and heat sensitize agent.

The aims of this study are to produce latex elastic ligatures and to compare the percentages of force degradation and the percentages of permanent deformation between latex and clear module non-latex elastic ligatures in 37 °C synthetic saliva.

1.3 Objectives

This experimental study aims to produce elastic ligatures made from Thai natural rubber and to compare their properties to the imported non-latex elastic ligatures. The properties to be tested will be force degradation and permanent deformation

CHAPTER 2

MATERIALS AND METHODS

2.1 Chemical and material

2.1.1 Chemicals and solvents

- 60% High ammonia concentrated latex (Chalong latex industry Co., Ltd., Thailand)
- 40% Formaldehyde solution (Lab scan analysis science Co., Ltd., Thailand) for adjusting pH of compound latex.
- 15% Terric (Polymer innovation Co., Ltd., Thailand) is stabilizer agent.
- 50 % Sulphur (S) (Lucky four Co., Ltd., Thailand) is vulcanising agent.
- 50% Lovinox CPL (Lucky four co., Ltd., Thailand) is antioxidant agent.
- 50% Zine-N-diethyldithuocarbamate (ZDC) (Lucky four Co., Ltd., Thailand) is accelerator agent.
- 50% Zine salt of 2-mercaptobenzothiazole (ZMBT) (Lucky four Co., Ltd., Thailand) is accelerator agent.
- 50% Zine Oxide (ZnO) (Lucky four Co., Ltd., Thailand) is activator agent.
- 10% Polyvinyl methylether (PVME) (V.I.P. interchem Co., Ltd., Thailand) is heat sensitive agent.
- Chloroform ($CHCL_3$)(J.T. Backer Inc. Co., Ltd. USA)
- Synthetic saliva (Faculty of dentistry, Prince of Songkla University)

2.1.2 Instruments

- Former of rubber sheet for tensile testing is a stainless steel 12x13x0.25 cm.
- Former of elastic ligatures is stainless steel wire diameter 1.2 mm.
- pH meter model pH 900 (Precisa instrument company, Switzerland)
- Hot air oven model 100-800 (Memmert GmbH + Co. KG, Germany)
- Brookfield viscometer model RVDV II+ (Viscometer Brookfield Engineering Inc., USA)

- Electric balance model 1212MSCS (Precisa instrument company, Switzerland)
- Universal tensile testing machine model LRX + (Ametex Inc, USA)
- Universal tensile testing machine model 1000Ss (Tensometric Co., LTD. England)
- Stereoscopic microscope model SMZ1500 (Nikon coperation, Japan)
- Stainless steel dowel (diameter = 4.0 mm.)
- Refrigerator
- Thermometer
- Hotplate and stirrer
- Latex compound stirrer

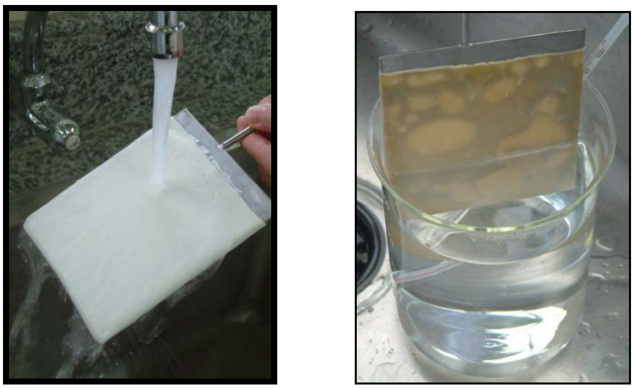
2.1.3 Samples

The imported elastomeric ligatures from Unitek® (Alastic A1 clear module) and Thai made elastic ligatures produced from the natural rubber in Prince of Songkla University (outside diameter 3.2 mm, inside diameter 1.2 mm and wall-thickness 1.0 mm) will be studied. All samples for each group are 60 samples for force degradation and permanent deformation experiments, 30 samples for each experiment.

2.2 Methodology

Latex elastic ligatures was prepared using concentrated latex filled with stabilizer (Terric), antioxidant (Lovinox CPL), activator (Zinc oxide), vulcanizing agent (Sulphur) and accelerator (Zinc diethyldithiocarbamate and ZMBT). Natural rubber latex concentration mixed with chemical agents was called latex compound. Latex compound was matured by stirring the latex compound using stirrer for 16 hours at room temperature. After maturing of the latex compound for 16 hours, they were tested the vulcanization level with chloroform testing (proper vulcanization level is at number 2 of chloroform testing). After vulcanization level testing, heat sensitizer agent (Polyvinyl methyl ether) was filled, then stored for 1 hours at 20 $^{\circ}$C. Later, acid-base value was adjusted to pH 8 with 10% formaldehyde at 20 $^{\circ}$C. Thereafter, the viscosity value was measured with Brookfield viscometer, at 100 RPM speeds with the axle spindle, for the valve of 2. A stainless steel former (12x13x0.25 cm) was heated at 100 $^{\circ}$C in a hot air oven and dipped into heat sensitive compound ten seconds for rubber thickness of 1.0 mm (Figure 1), to make a piece of rubber sheet. Dry film for five minutes at room temperature and the rubber sheet was leached with water at room temperature (wet gel leaching) to removed soluble protein (Figure 2A). The rubber sheet was vulcanized at 115 °C in a hot air oven for 50 minutes and later, then the dry film was leached with water at 70 °C for 15 minutes and vulcanized again at 115 °C for 5 minutes (Figure 2B).

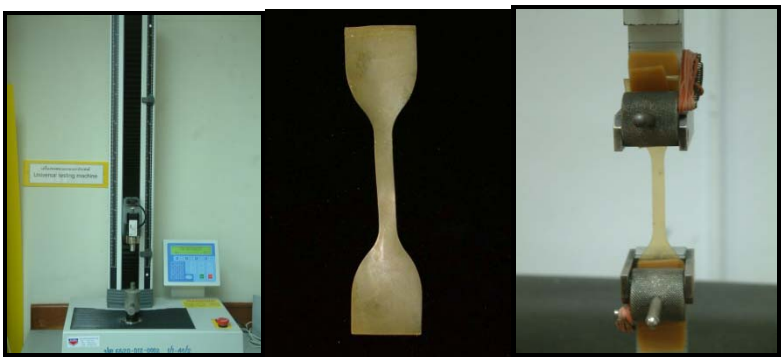
Fig 1. Stainless steel former dipped into heat sensitive compound for rubber thickness of 1.0 mm.



A B

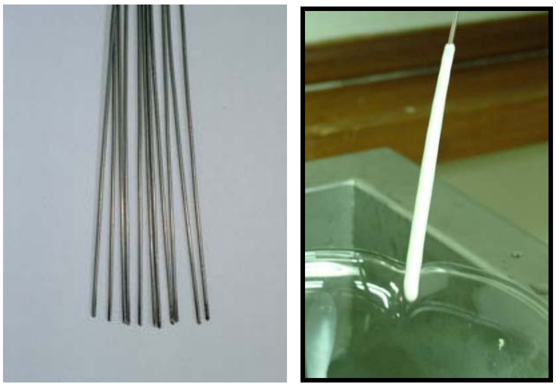
Fig 2. A: Wet gel leaching with water at room temperature; B: Dry film leaching with water at 7 °c.

The rubber sheet was cut with die C into dumbbell shape 1.0 mm thickness for tensile strength testing with a Universal testing machine using 50 kN load cell with a cross-head speed of 500 mm/min (Figure 3A, 3B and 3C). The compound had adjusted until tensile strength was accomplished (at least 22 MPa). This proportion of vulcanizing agent and accelerator in latex compound was used to produce latex ligatures (Thai latex elastic ligatures).



A B C

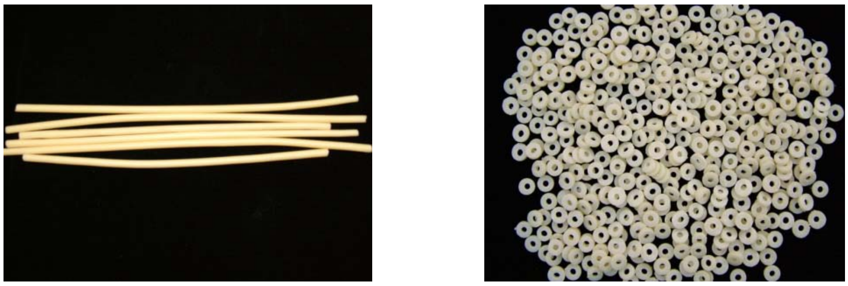
Fig 3. A: Universal testing machine (Tensometric Co., LTD. England); B: Dumbbell; C: Dumbbell latex sample was tested for tensile strength.



A **B**

Fig 4. A: A stainless steel wire former (diameter 1.2 mm); B: Dipped into heat sensitive compound.

When, latex compound formula has already, latex elastic ligatures were made by heat sensitive dipping in latex compound, using a mold made from 1.2 mm diameter of a stainless steel wire former (equal to the internal diameter of imported elastic ligatures in this study)(Figure 4A). The mold was dipped into latex compounds to form a thickness approximately 1 mm (Figure 4B). The rubber tube was processed with wet gel and dry film leaching. The rubber tube was leached with water at room temperature (wet gel leaching) to remove soluble protein before vulcanizing, then the dry film was leached with water at 70^{o}C. The latex elastic tubes (Figure 5A) were cut for proper the dimension of latex elastic ligatures. (Figure 5B) (1.2 mm of inside diameter (ID), 3.2 mm of outside diameter (OD), 1.0 mm of wall thickness (WT) and 0.8 mm. of the width) (Figure 6)



A B

Fig 5. A: The elastic tubes; B: The elastic ligatures.

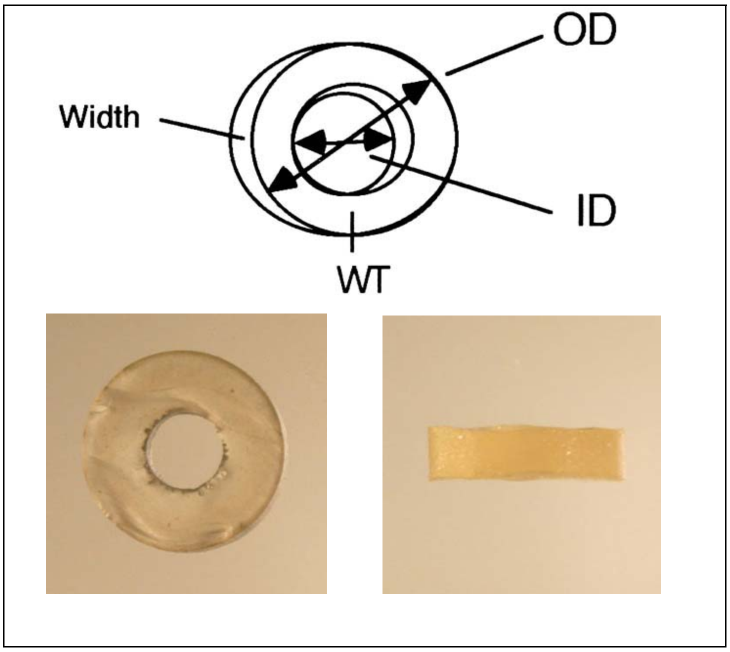


Fig 6. A ligature in three dimensions.

The groups of elastic ligatures tested in this study including clear molded elastomeric ligatures from 3M Unitek® (UNI) and latex elastic ligatures from Prince of Songkla University (LE). Latex elastic ligature samples were prepared to have the same size as UNI. Samples from each group were divided into two tested groups. Thirty samples from each group were studied in simulated oral environment for force degradation and thirty samples from each group were studied in simulated oral environment for permanent deformation.

Before testing, the initial wall thickness, inside diameter, and outside diameter of all ligatures in test groups were measured with Stereoscopic microscope (Nikon, model smz1500, Japan)(figure 7). All elastic ligatures were stretched over stainless steel dowels (4.0 mm. of diameter) to simulate the stretch necessary to apply an elastomeric ligatures over a maxillary central incisor twin bracket (0.022 inches slot) and arch wire (Figure 8). All elastic ligatures were stored in synthetic saliva at 37^{O}C to simulate an oral environment.

Fig 7. Stereoscopic microscope (Nikon, model smz1500, Japan) and a picture of a ligature during dimensional measurement.

Fig 8. Ligatures were stretched over stainless steel dowel.

Group 1. Force degradation experiment

The forces of elastic ligatures were measured using a LIOYD II Instron testing machine with a load cell of 10 N (Ametex Inc., model LRX+,USA)(figure 9) and recorded after the initial activation; 24 hours, 7 days, 14 days, and 28 days. All elastic ligatures were stretched in the testing machine to measure the force magnitude with stretched velocity of 0.2 inches/minute at range 5.5 mm between two hooks made of 0.45 mm diameter stainless steel wire fixed to the testing machine. The results were calculated for the mean percentage of force degradation for each time period.

The percentage of force degradation was obtained from each specimen as follows:

$$\text{Percentage of force decay} = \frac{Fo - Ft}{Fo} \times 100$$

Where

Fo: initial force

Ft: force at times (24 hours, 1 week, 2 weeks and 4 weeks)

A

B

Fig 9. A: Universal tensile testing machine model LRX + (Ametex Inc, USA) with a load cell of 10 N. B: The ligature was stretched in the testing machine to measure the force.

Group2. Permanent deformation experiment

After initial measurements, samples were placed into the synthetic saliva bath at 37^{o}C for 28 days. At 28 days, the samples were removed, and the WT, ID, and OD were measured to evaluate to effect of water sorption on the dimensions of the elastic ligatures. The results were calculated for mean percentage of changes for each elastic ligature dimensional measurement.

From each specimen, the percentage of permanent deformation was obtained as follows:

$$\text{Percentage of permanent deformation} = \frac{Do - Df}{Do} \times 100$$

Where

Do: initial dimension

Df: dimension at 4 weeks

Mean dimensional differences from initial measurements to final measurements from each group were compared using paired *t* tests. The percentages of force degradation for each group were evaluated by repeated measures analysis of variance (ANOVA) at different times. Independent paired t- test was used to compare the mean percentage of dimension change and mean percentage of force degradation between groups in the same dimension and same time. Correlation coefficients were calculated to determine the relationship between the percentage of force degradation and initial force level and the percentage of permanent deformation at the initial and after 28 days stretching period.

CHAPTER 3

RESULTS

At the preliminary stage, formula, pH and viscosity of latex compound from Dumbbell latex samples examined in the Universal testing machine after adjusting the composition until the proper tensile strength was accomplished and used for producing latex elastic ligatures were shown in table 1. Table 2, the tensile strength of this formula was 24.54 mega Pascal (at least 22 mega Pascal) presented with the amount of sulphur and the thickness of the rubber dumbbell sheets.

Table 1. Latex and chemicals content of latex compound. (phr)

Latex and chemicals Content.	Formula (phr)
60% HA Latex	100
15% Terric	0.50
50% Lovinox CPL	1.0
50% Sulphur	3.0
50% ZDC	1.0
50% ZMBT	0.5
50% ZnO	0.5
10% PVME	1.0
pH	8.55
Viscosity (cps)	265

In second stage, the mean initial dimensions (WT, ID, OD) of the ligatures in groups 1 and 2 are presented in tables 3 and figure 10. UNI, is smaller in ID and OD than the manufacturer's specifications. All means of the initial dimensional measurements of LE showed significantly larger than UNI's means. However LE has very larger than UNI in all dimensions. (0.02 mm for WT, 0.08 mm for ID, and 0.12 mm for OD)

Table 2. The thickness, tensile strength and elongation at break of the rubber sheets.

Amount of Sulphur (phr)	Thickness (mm)	Tensile Strength (MPa)	Elongation @Break(%)
3.00	1.06 (0.051)	24.54 (0.460)	715 (13.693)

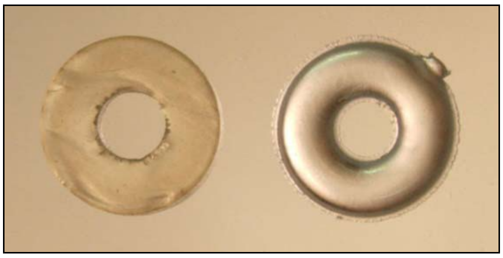


Fig 10. Initial dimension of latex elastic ligatures and elastomeric ligatures.

Table 3. Manufacturer, brand name, and measurements wall thickness (WT), inside diameter (ID) and outside diameter (OD). (* significant between groups at 0.05 level)

Type	Brand name	WT (mm.)	ID (mm.)	OD (mm.)
Unitek® (UNI)	Alstik A1 module	1.05$\pm$0.01*	1.15$\pm$0.02*	3.25$\pm$0.02*
PSU (LE)	Thai latex elastic ligature	1.07$\pm$0.03	1.23$\pm$0.03	3.37$\pm$0.05

Table 4 and figure 11 show initial, 24 hours, 7, 14 and 28 days of force levels for groups 1. At all times, LE shown significant lower the force level compared to UNI. At initial and final times, LE (162.93 g, 116.86 g) has significantly lower force level than UNI (702.14 g, 194.51 g). Both materials had a decrease in force over time. The rate of force degradation was greatest in the first day. After that the force continued to decease gradually with very slow rate. In Figure 11, force-time curve of elastic ligatures is demonstrated, where it is evident that the curve could be separated in two distinct components: a steep slope at initial period and a low inclined slope part at subsequence period. The first, which represents a rapid force loss, seems to take place within the first day after extension. The mean percentage of force degradation of the materials included in the study is shown in table 5 and figure 12. LE has significantly lower the percentage of force loss (16.30 %) compared to UNI (66.07 %) from initial to 24 hours. The second components present an almost stable force with gradually force reduction from 1 day to 28 days. Throughout the testing period, a significant differences in percentage of force degradation between LE (28.26%) and UNI (72.31%). At all time, both materials showed significant increase the percentage of force degradation with time and LE has little gradually force loss (28.26 %) compared to UNI (72.31 %). At initial time, the coefficient of variations of force level of both UNI and LE were 7.76% and 6.41%, respectively.

Table 4. Mean force values (g) of non-latex elastic ligatures (UNI) and latex elastic ligatures (LE) at 5 times comparisons.

Type	Simple	Mean and standard deviation of force level (gram)				
	N	Initial	1 day	7 days	14 days	28 days
UNI	30	702.14±54.52	236.71±9.02	223.34±18.53	196.92±16.72	194.51±17.03
LE	30	162.93±10.45	136.33±8.64	124.92±9.85	119.55±8.66	116.86±7.96

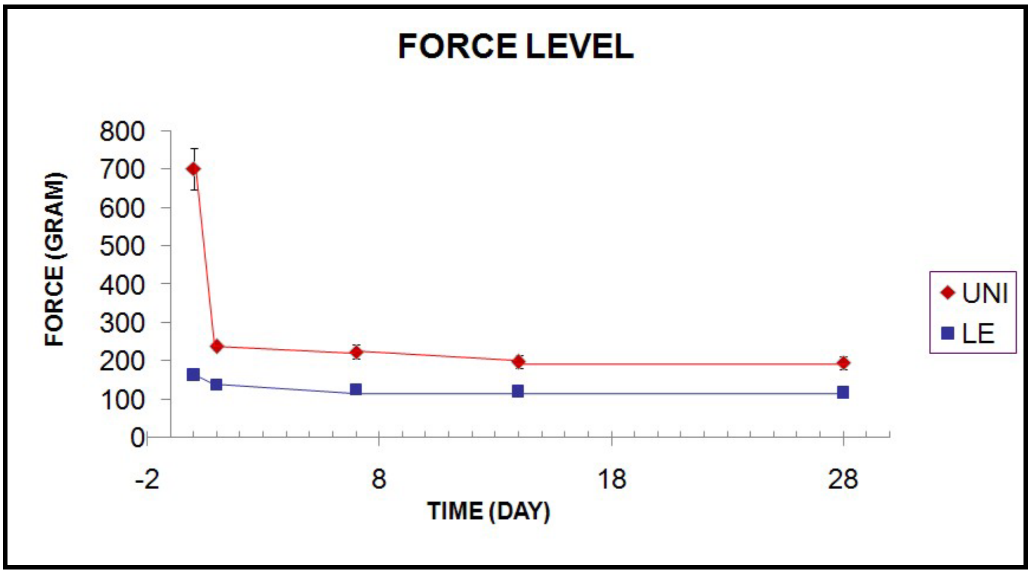


Fig 11. Mean and SD of force level of non-latex elastic ligatures (UNI) and latex elastic ligatures (LE) in 37°C synthetic saliva.

Table 5. Mean and SD percentage of force degradation of non latex elastic ligatures (UNI) and latex elastic ligatures in 37°C synthetic saliva. (* significant with in group, ** significant between groups)

Type	Simple	Mean and standard deviation of loss of force (%)			
	N	1 day	1 wk	2 wks	4 wks
UNI	30	66.07±3.21*	68.19±0.87*	71.96±0.65*	72.31±0.74*
LE	30	16.30±1.83* **	23.35±3.12* **	26.64±1.85* **	28.26±2.14* **

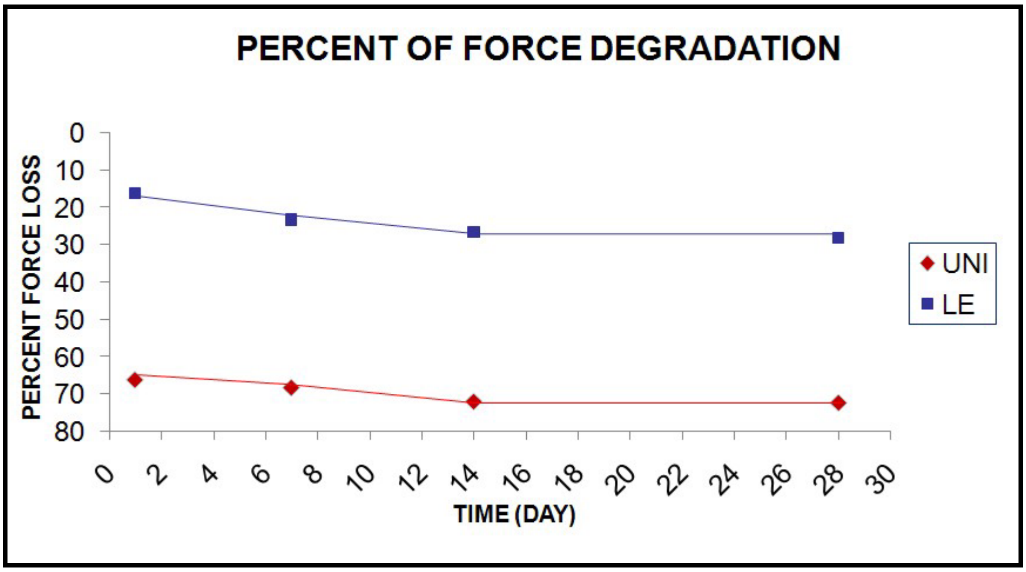


Fig 12. Mean and SD percentages of force degradation of non latex elastic ligatures (UNI) and latex elastic ligatures (LE) in 37°C synthetic saliva.

The table 6 and figure 13-15 show the mean initial and final dimensional measurements for groups 2 that all elastic ligatures were significantly different after the tests (Tables 6 with independent paired t test comparisons). Figure 16 shows the elastic ligatures between testing periods. The amount of permanent deformation was greater in UNI than in LE. In table 7 and figure 15, LE shows significantly lower percentage of permanent deformation than UNI in all dimensions. In WT, LE (-2.89%) has statistically significant lower percentage of permanent deformation than that of UNI (-44.10%). In ID, LE (-24.34%) has statistically significant lower percentage of permanent deformation than that of UNI (191.30%). In OD, LE

(6.97%) has statistically significant lower percentage of permanent deformation than that of UNI (38.90%).

Table 6. Test group 1; mean dimensions, mean percentage change for WT, ID, OD at constant stretch in salivary bath at 37°C for 28 days with independent pair t-test (P≤0.05) comparing percentage of dimension change between types. (* significant with in group, ** significant between group)

Type	Sample	Initial dimensions (mm.)			Final dimensions (mm.)			Mean % change		
	N	WT	ID	OD	WT	ID	OD	WT	ID	OD
UNI	30	1.05±0.01	1.15±0.02	3.25±0.02	0.59±0.03 *	3.34±0.05 *	4.51±0.06 *	-44.10 **	191.30 **	38.9 **
LE	30	1.07±0.03	1.23±0.03	3.37±0.05	1.04±0.04 *	1.53±0.04 *	3.60±0.07 *	-2.89	24.34	6.97

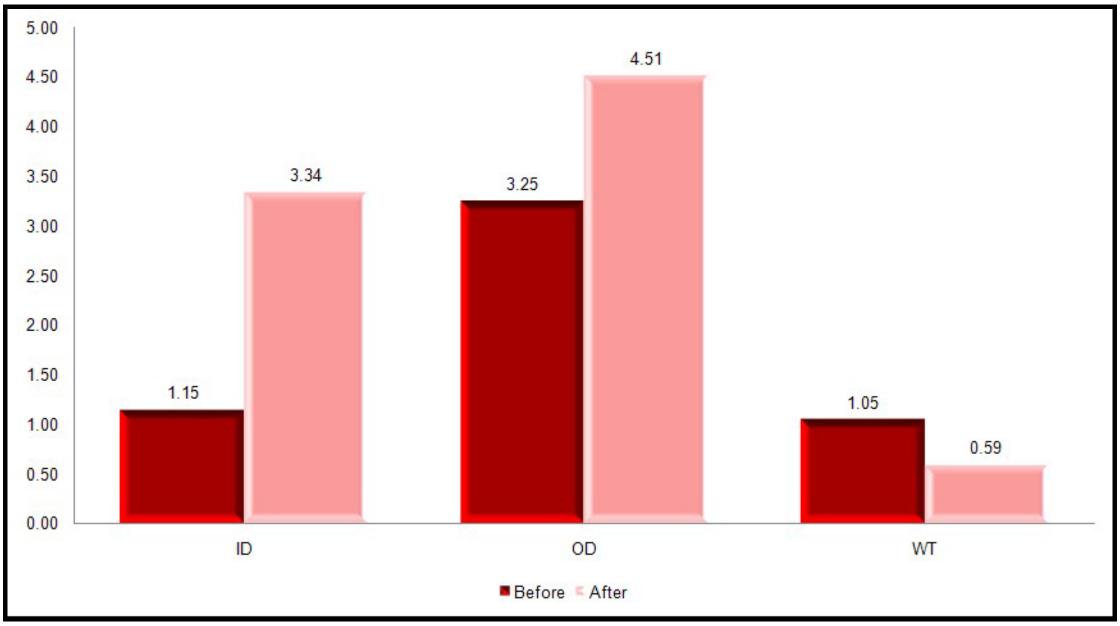


Fig 13. All dimension of UNI before and after 28 days.

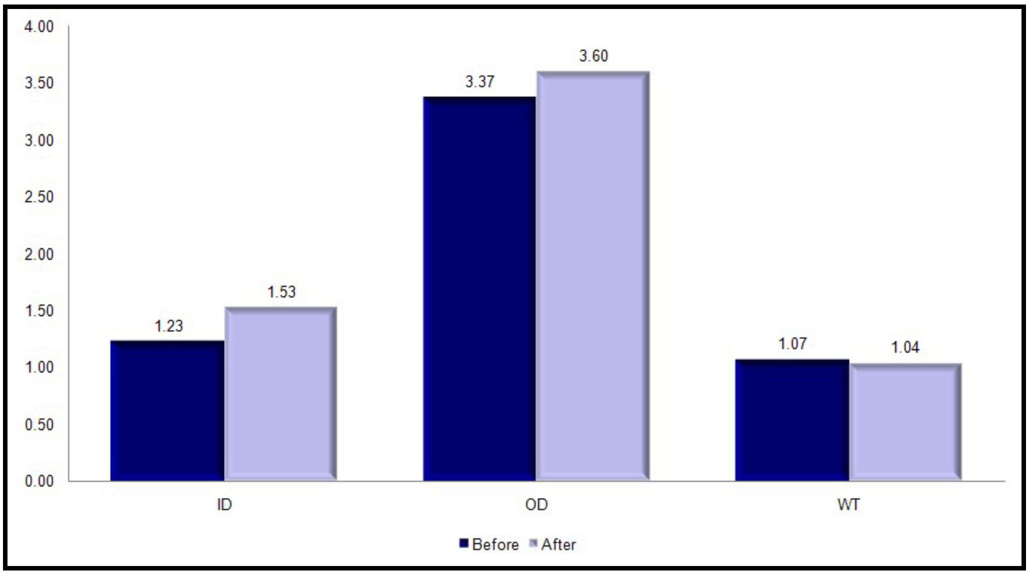


Fig 14. All dimension of LE before and after 28 days.

Table 7. Mean and SD percentages of permanent deformation in all dimensions of non-latex elastic ligatures (UNI) and latex elastic ligatures (LE) in 37°C synthetic saliva.

Elastic ligature	% permanent deformation		
	WT	ID	OD
UNI	-44.10±2.91	191.30±5.45	38.90±1.57
LE	-2.89±4.52	24.34±4.50	6.97± 2.38

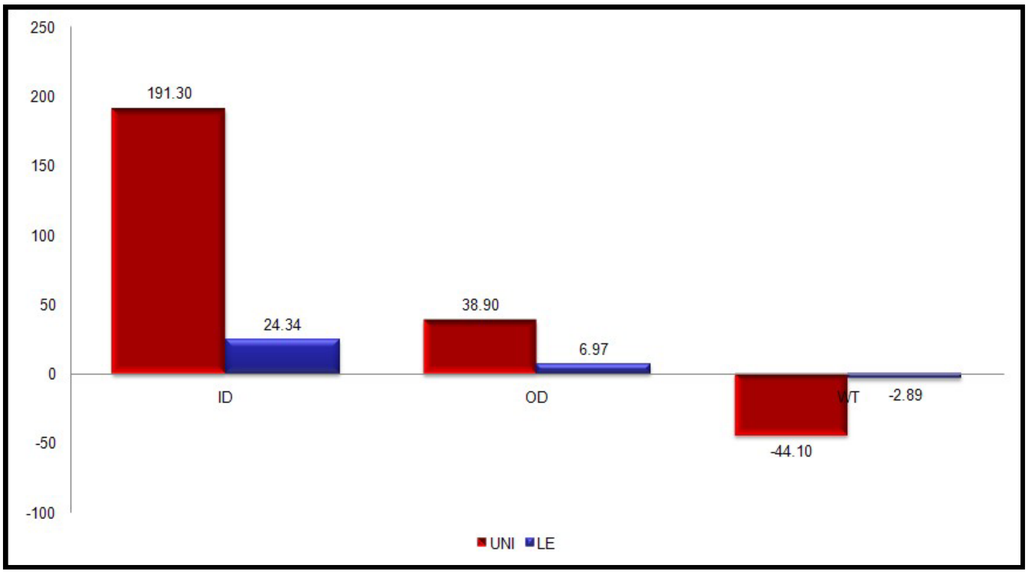


Fig 15. Compare percentage of permanent deformation UNI with LE.

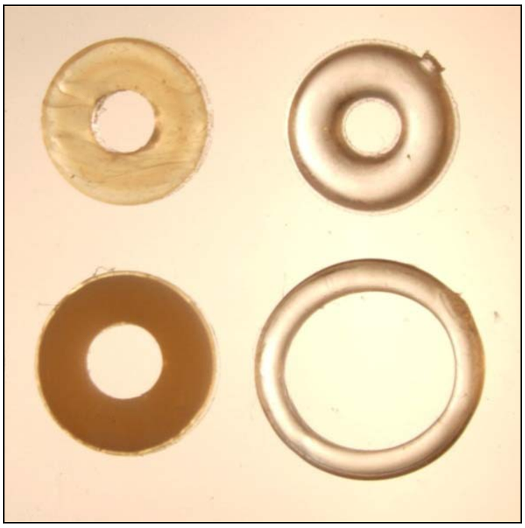


Fig 16. Samples after 4 weeks testing (bottom row) and equivalent untested sample (top row) (left to right) LE and UNI.

Both of ligature types in testing, correlation analysis demonstrated no significant associations between the percentage of force degradation and initial force level, percentage of inside diameter change, percentage of outside diameter change and percentage of wall thickness change, as shown in Table 8.

Table 8. Correlation coefficients between the percentage of force degradation and initial force level, % wall thickness change, % inside diameter change, % outside diameter change.

Type	Number	Parameter	r	Significance
UNI	30	Initial force	-0.22	NS
		% Wall thickness change	-0.047	NS
		% Inside diameter change	0.09	NS
		% Outside diameter change	0.132	NS
LE	30	Initial force	0.101	NS
		% wall thickness change	-0.021	NS
		% inside diameter change	0.244	NS
		% outside diameter change	-0.064	NS

NS, not significant

Both rubber elastic ligatures and elastomeric ligatures underwent some changes in their appearance at the end of the investigation. Rubber elastic ligatures become less transparent or rather opaque and turned from a yellowish straw color to a rather white color.

CHAPTER 4

DISCUSSION

This study intended to test the force degradation of elastomeric ligatures correlated to timing by stretching the ligatures on round dowel instead of rectangular jig which has the same shape as brackets. The round dowel was selected rather than rectangular jig due to the following reasons. First, the ligature shapes between elastomeric and natural rubber after stretching are different when the rectangular jig is used. The elastomeric ligature is changed to be square shape while the natural rubber ligature still maintains its round shape. This shape difference makes the measurement after stretching not possible to compare. Second, after the shape changed, the points where the hooks are placed to measure force of the elastomeric ligature in the testing machine will always slip to the corners which are much narrower than the sides of the rectangular. These corners are probably weaker than that could affect the measurement of the forces. Compared to the natural rubber ligature, the shape after stretching is perfect circle so the thickness of the ligature is even. The points where the hooks are placed would have the same thickness so the force from the measurement will not vary.

In this study the force degradation was recorded in 24 hours, 1 week, 2 weeks and 4 weeks for investigating behavior of the both elastic ligatures. As a result of the previous report[12] showed the most of the force degradation of elastomeric ligatures occurred within the first day and continued at a slower rate during the rest period and in clinical, patients were recalled for routine visit with a mean time interval of 4 weeks.

In this study, UNI was representative of the several available elastomeric ligatures in the market to compare with our new product because our pilot study found that UNI had less permanent deformation than other brands in the first day of testing. (Energy, Ortho Organizer, W&H, Dynaflex)

The media in which elastics had been tested from prior studies was varied and considerably effect to the results. For instance, Andreasen and Bishara[18] carried out experiments in dry and simulated oral environments of 100% humidity conditions and reported no significant differences for the different conditions. Ash and Nikolai[21] and Ju Hwang[22] stated that greater force degradation was observed in wet condition than dry conditions of the same temperature. Taloumis et al[12] showed elastomeric ligatures were affected by moisture and heat, exhibited rapid force loss (53% to 68% in 24 hours), and deformed permanently when stretched. This study is the first in vitro behavior comparing of both elastic ligatures produced by differences type. An attempt to control some variables (pH of artificial saliva, temperatures, time interval, stretch velocity) the influence the vitro behavior of the elastic ligatures was made to imitate the oral environment.

In this study, Table 4 and figure 11 showed initial, 24 hours, 7, 14 and 28 days of force levels for groups 1. At all times, LE showed significant lower the force level compared to UNI. At initial and final times, LE (162.93 g, 116.86 g) has significantly lower force level than UNI (702.14 g, 194.51 g). Both materials exhibited decreased in force over time. The rate of force degradation was greatest in the first day. After that the force continued to decease gradually with very slow rate. In Figure 11, force-time curve of elastic ligatures is demonstrated, where it is evident that the curve could be separated in two distinct components: a steep slope at initial period and a low inclined slope part at subsequence period. The first component, which represents a rapid force loss, seemed to take place within the first day after extension but UNI force levels were rapidly decreased than LE force levels within the first day. It is suspected that UNI has high initial force in order to compensate the subsequence force loss. Moreover, the results of coefficient of variations showed that the variation of UNI initial force is higher than that of LE which are 7.76%, 6.41%, respectively.

In this study, according to the variability of initial force values, the percentage of force degradation comparing to initial force was used rather than actual generated force. The pattern of the percentage of force degradation of both materials in this study was similar to the previous studies. The percentages of force degradation of LE in this study were approximately 16.3%, 23.35%, 26.64% and 28.26% for 24 hours, 1 week, 2 weeks and 4 weeks, respectively. Those were similar to previous studies of natural latex elastic. Bishara and Andreasen[15] found that the percentages of force degradation of latex elastic bands in water at room temperature were approximately 17.2 %, 21.9% and 32.5% for 24 hours, 1 week and 3 weeks, respectively. Thanagornjuk[23] found that the percentage of force degradation of Thai-made elastic chains in 37°C distilled water at variously stretched lengths (20 mm and 25 mm) were 26.49-26.80% after 4 weeks. Suvapap[24] found that the percentages of force degradation of latex elastics band held at 20 mm extension for 1 day were approximately 16.03% and 15.77% for G&H® and Thai-made elastic bands, respectively. After 1 day, the percentage of force degradation of all elastic bands was smaller when continue to stretch for 4 weeks. Kersey et al[16] found that the percentage of force degradation of American Orthodontics® latex elastic bands of 0.25 inch, 4.5 oz was 17% at 24 hours.

In this study, the percentages of force degradation of UNI were approximately 66.07%, 68.19%, 71.96% and 72.31% for 24 hours, 1 week, 2 weeks and 4 weeks, respectively. Resemble to previous studies about polyurethane, Thanagornjuk[23] found the percentages of force degradation of non-latex elastic chains from Unitek® in 37°C distilled water at variously stretched lengths (20 mm, 25 mm and 30 mm) were 63.78-65.51% after 4 weeks but the test was done in distilled water. Bishara and Andreasen[15] found that the percentages of force degradation of non-latex elastic bands in water at 35 °C were approximately 54.7 %, 60.5% and 74.9% for 24 hours, 1 week and 3 weeks, respectively. Kersey et al[16] compared 1/4 inch latex and non-latex elastic bands from the same manufacturer and measured those elastic bands at 24 hour in water at 37°C by cyclic testing, they found that the force degradation in non-latex elastic bands was 47%. Taloumis et al[12] found the percentage of force degradation of molded gray elastomeric ligature from UNI averaged 68% during the first day and increased to 78% at 4 weeks which are comparable to this study that found the force degradation of UNI averaged 66% during the first day and increased to 72% at 4 weeks.

In this study, the clear UNI produced initial force of an average of 702 grams with approximately 60 % and 72 % force degradation at 24 hours and 4 weeks respectively. Unfortunately, up to now, there are only one study examining elastomeric ligatures and that studied ligatures were grey elastomeric ligatures. Taloumis et al[12] found that the grey UNI produced less initial force of 548 grams and higher percentage of force degradation of approximately 68 % and 78 % for 24 hours and 4 weeks. The comparison between our study and Taloumis et al[12]'s study shows the difference as a result of the different materials of our clear and their grey elastomeric ligatures similar to Lu et al[25]' s elastic chains study. Lu et al[25] compared the force degradation properties of short filament grey and clear chains, the clear chains generally provided a higher initial force level and retained a larger percentage of this force while extended at a constant length and stored for 1 week in fluid environment.

In our study, no relationship was found between the initial force and the amount of force degradation in UNI and LE (table 8) supported by Hershey and Reynolds[26]'s study and

Suvapap's study, respectively. However, De Genova et al[27] found that elastomeric chains producing higher initial forces displayed less force loss than did chains with lower initial forces. Contrast to Lu et al[25], study which reported that the greater the initial force of the elastomeric chain, the greater the amount of force degradation increases.

In our study, from Table 8, both of elastic ligature types showed no correlation between % WT, ID and OD permanent deformation of the modules and the percentage of force degradation.

The method in this study may suffer and probably more critical weakness relates to the excessive handling of the specimens and repeated extensions of the same specimen at different time intervals to record force loss. This process may induce fatigue of the material, precluding a reliable extrapolation of the extent of relaxation. Same in Taloumis et al[12]study, who reported the elastomeric ligatures test force at initial and 28-day period (33.50%) have more remaining force than the elastomeric ligatures test force interval at 24 hours, 7 days, 14 days, and 28 days (21.35%).

All of the results showed that LE had lowered the percentages of force degradation and permanent deformation. In synthetic elastic, such as elastomers, the force arises from the macromolecular chain entanglements, ie, in interconnection of chains. On the contrary, in natural rubbers such as latex, the retracting force is because of the covalent bonding and cross-linking of chains. The differences in structure between non-latex elastic ligatures and latex elastic ligatures can explain the differences in properties that were reported in this study.

In the salivary environment of this study, the latex elastic ligatures underwent some changes in appearance. In the latex elastic ligatures (LE), the color changed from a yellowish straw to off-white, the appearance was swollen, opaque and slightly permanently deformed. In the elastomeric ligatures (UNI), the colors are not changed, still translucent but more permanently deformed.

Reitan[28] believed that to obtain fairly rapid tooth movement, hyalinization zones were to be avoided or kept to a continuous forces. Proffit[29] recommended Initial arch wires for alignment should provide light, continuous force of approximately 50 grams, to produce the most efficient tipping tooth movement. On the contrary, heavy force should be avoided. In this study, LE showed the force values throughout testing approximately 163-117 grams. This force level from LE should hold an arch wire in the bracket slot when used in the initial alignment and leveling phases of orthodontics. Furthermore, Bennett JC and McLaughlin RP[6] suggested applying use elastomeric ligatures for closing space or call that “elastic tiebacks”. From Samuel clinical study[9, 30], the light continuous forces was made the tooth move faster than the heavy intermitted force.

Further study should be performed for testing the biocompatibility, the cytotoxicity test is necessary to prove biocompatibility because latex elastic ligatures are kept in the closed space of the oral cavity for several days. Moreover the future study should be performed in clinic and focused on stress-strain, relaxation (cyclic test) and density of materials.

CHAPTER 5

CONCLUSIONS

1. The percentages of force degradation and permanent deformation of the latex elastic ligatures were statistically significant less than the elastomeric ligatures (Unitex®) at every experimental timing interval period**.**
2. Both of elastic ligature types showed no correlation between % WT, ID and OD permanent deformation of the modules and the percentage of force degradation.
3. Both of elastic ligature types showed no correlation between the initial force values and the percentage of force degradation of the modules.
4. The latex elastic ligatures produced at Prince of Songkla University have potential to apply during initial aligning and leveling for possible better tissue response.

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APPENDIX

1. **Data of initial dimensions of elastomeric ligatures (Unitek®) in force degradation experiment.**

Number	Dimension (mm.)		
	Wall thickness	Outside diameter	Inside diameter
1	1.08	3.26	1.11
2	1.07	3.27	1.13
3	1.06	3.25	1.14
4	1.07	3.25	1.11
5	1.07	3.25	1.11
6	1.06	3.23	1.11
7	1.06	3.25	1.14
8	1.04	3.29	1.22
9	1.07	3.29	1.16
10	1.08	3.27	1.11
11	1.04	3.21	1.14
12	1.05	3.22	1.13
13	1.06	3.26	1.14
14	1.06	3.26	1.14
15	1.07	3.26	1.12
16	1.06	3.26	1.14
17	1.08	3.27	1.12
18	1.07	3.24	1.11
19	1.07	3.25	1.12
20	1.07	3.26	1.13
21	1.06	3.25	1.13
22	1.08	3.26	1.11
23	1.06	3.25	1.14
24	1.07	3.24	1.11
25	1.06	3.25	1.14
26	1.08	3.27	1.12

Number	Dimension (mm.)		
	Wall thickness	Outside diameter	Inside diameter
27	1.06	3.25	1.13
28	1.06	3.26	1.14
29	1.07	3.27	1.14
30	1.07	3.27	1.14

2. Data of initial dimensions of latex elastic ligatures in force degradation experiment.

Number	Dimension (mm.)		
	Wall thickness	Outside diameter	Inside diameter
1	1.09	3.37	1.20
2	1.10	3.40	1.21
3	.97	3.17	1.23
4	1.07	3.37	1.24
5	1.08	3.39	1.23
6	1.00	3.21	1.21
7	1.13	3.48	1.23
8	1.00	3.27	1.27
9	1.10	3.41	1.22
10	1.10	3.43	1.24
11	1.07	3.36	1.23
12	1.05	3.36	1.27
13	1.08	3.30	1.14
14	1.10	3.39	1.20
15	1.08	3.38	1.23
16	1.02	3.20	1.17
17	1.14	3.49	1.21
18	1.05	3.32	1.22
19	1.09	3.42	1.25
20	1.12	3.43	1.20
21	1.06	3.36	1.24
22	1.04	3.31	1.23
23	1.12	3.44	1.21
24	1.10	3.40	1.21
25	1.07	3.37	1.24
26	1.03	3.27	1.21
27	1.16	3.49	1.18

Number	Dimension (mm.)		
	Wall thickness	Outside diameter	Inside diameter
28	1.05	3.32	1.22
29	1.12	3.44	1.21
30	1.13	3.48	1.22

3. Data of initial dimensions of elastomeric ligatures (Unitek®) in permanent deformation experiment.

Number	Dimension (mm.) initial		
	Wall thickness	Outside diameter	Inside diameter
1	1.07	3.26	1.13
2	1.02	3.22	1.18
3	1.04	3.23	1.16
4	1.05	3.22	1.12
5	1.06	3.24	1.13
6	1.06	3.24	1.12
7	1.04	3.25	1.17
8	1.05	3.25	1.16
9	1.04	3.25	1.17
10	1.04	3.24	1.16
11	1.04	3.24	1.16
12	1.06	3.23	1.12
13	1.06	3.26	1.14
14	1.04	3.22	1.14
15	1.04	3.23	1.16
16	1.05	3.23	1.14
17	1.07	3.29	1.16
18	1.04	3.24	1.16
19	1.05	3.23	1.14
20	1.06	3.24	1.13
21	1.07	3.28	1.14
22	1.05	3.23	1.13
23	1.07	3.25	1.12
24	1.05	3.23	1.14
25	1.07	3.27	1.13
26	1.06	3.26	1.14

Number	Dimension (mm.)		
	Wall thickness	Outside diameter	Inside diameter
27	1.09	3.30	1.12
28	1.05	3.24	1.15
29	1.07	3.26	1.13
30	1.04	3.29	1.21

4. Data of initial dimensions of latex elastic ligatures in permanent deformation experiment.

Number	Dimension (mm.) initial		
	Wall thickness	Outside diameter	Inside diameter
1	1.05	3.34	1.25
2	1.08	3.37	1.21
3	1.10	3.45	1.26
4	1.10	3.41	1.21
5	1.03	3.34	1.29
6	1.02	3.26	1.23
7	1.01	3.26	1.25
8	1.10	3.40	1.20
9	1.09	3.41	1.23
10	1.05	3.33	1.23
11	1.11	3.40	1.19
12	1.11	3.46	1.24
13	1.10	3.40	1.20
14	1.07	3.33	1.20
15	1.11	3.42	1.20
16	1.02	3.35	1.31
17	1.10	3.41	1.21
18	1.04	3.29	1.21
19	1.07	3.40	1.26
20	1.04	3.29	1.22
21	1.09	3.38	1.21
22	1.05	3.29	1.20
23	1.03	3.32	1.26
24	1.11	3.42	1.20
25	1.10	3.40	1.21
26	1.02	3.33	1.29

Number	Dimension (mm.)		
	Wall thickness	Outside diameter	Inside diameter
27	1.09	3.38	1.20
28	1.11	3.41	1.19
29	1.06	3.37	1.26
30	1.10	3.39	1.20

5. Data of force level (gram) of elastomeric ligatures (Unitek®) in force degradation experiment at initial, 24 hours, 1 week, 2 weeks and 4 weeks.

Number	Force level (gram)				
	Initial	24 hours	1 week	2 weeks	4 weeks
1	689.93	238.32	218.05	195.28	192.80
2	759.21	244.62	233.66	212.65	211.12
3	791.82	237.91	252.53	228.06	226.99
4	640.96	245.86	199.62	177.97	175.32
5	746.50	243.42	236.50	207.02	207.79
6	701.04	240.43	221.94	197.54	195.51
7	692.17	246.54	218.33	193.20	188.39
8	750.25	253.64	247.90	220.60	217.38
9	783.61	239.93	247.45	222.40	213.25
10	620.17	247.70	196.73	171.74	170.01
11	632.80	240.85	216.91	183.03	178.95
12	681.60	248.58	219.23	188.04	184.39
13	660.58	223.86	214.75	181.66	180.24
14	622.28	227.81	184.22	168.32	167.40
15	757.98	226.08	230.84	205.55	203.67
16	735.98	232.75	235.73	205.45	201.37
17	765.15	232.72	248.84	222.45	218.64
18	729.98	226.49	237.69	207.52	208.80
19	709.06	228.81	229.21	205.34	206.60
20	684.78	231.34	218.22	193.20	198.61
21	771.66	226.02	249.80	218.59	215.99
22	660.06	233.07	207.66	179.89	178.68
23	778.12	220.90	243.15	210.12	210.84
24	722.35	239.48	234.71	201.60	198.24
25	711.30	216.66	221.63	199.30	197.19
26	679.39	244.76	218.09	191.20	185.25

Number	Force level (gram)				
	Initial	24 hours	1 week	2 weeks	4 weeks
27	665.16	234.91	217.35	184.32	181.00
28	614.92	242.56	195.95	173.79	168.00
29	611.85	238.91	189.32	176.14	172.58
30	693.57	246.44	214.29	185.68	180.17

6. Data of force level (gram) of latex elastic ligatures in force degradation experiment at initial, 24 hours, 1 week, 2 weeks and 4 weeks.

Number	Force level (gram)				
	Initial	24 hours	1 week	2 weeks	4 weeks
1	165.39	144.86	142.19	122.67	117.16
2	150.68	129.15	116.34	107.60	108.21
3	172.78	143.16	137.88	129.08	122.91
4	163.79	134.49	120.08	117.84	112.63
5	157.09	134.49	123.48	119.05	117.63
6	160.67	136.20	119.80	110.03	111.97
7	175.32	146.76	141.32	125.30	123.27
8	163.58	135.99	122.12	121.98	116.81
9	152.92	127.68	116.16	114.71	109.15
10	175.99	144.25	136.05	130.01	122.55
11	169.83	136.60	130.18	120.85	121.03
12	180.18	152.33	135.22	134.85	127.39
13	164.66	138.00	134.93	121.05	125.01
14	167.61	141.75	129.50	125.85	121.51
15	150.01	122.40	111.76	111.37	103.62
16	156.73	133.50	124.96	112.97	115.39
17	176.81	149.87	136.45	135.57	135.09
18	154.63	130.36	120.64	114.92	113.43
19	169.17	137.68	128.43	122.14	119.24
20	171.67	149.14	133.34	125.26	126.07
21	155.96	130.18	115.66	116.25	115.73
22	179.79	146.43	135.39	134.85	127.39
23	147.75	121.61	107.32	105.42	102.61
24	137.54	120.76	110.08	104.31	104.21
25	163.79	134.49	120.08	117.84	112.63
26	168.20	139.47	122.19	123.29	119.97

Number	Force level (gram)				
	Initial	24 hours	1 week	2 weeks	4 weeks
27	160.76	134.23	124.56	115.50	116.04
28	160.08	132.23	122.90	115.19	114.40
29	147.75	121.61	107.32	105.42	102.61
30	166.83	140.18	121.16	125.36	120.13

7. Data of percentage of force degradation of elastomeric ligatures (Unitek®) in force degradation experiment at 24 hours, 1 week, 2 weeks and 4 weeks.

Number	Percentage of force degradation (%)			
	24 hours	1 week	2 weeks	4 weeks
1	65.46	68.40	71.70	72.06
2	67.78	69.22	71.99	72.19
3	69.95	68.11	71.20	71.33
4	61.64	68.86	72.23	72.65
5	67.39	68.32	72.27	72.16
6	65.70	68.34	71.82	72.11
7	64.38	68.46	72.09	72.78
8	66.19	66.96	70.60	71.03
9	69.38	68.42	71.62	72.79
10	60.06	68.28	72.31	72.59
11	61.94	65.72	71.08	71.72
12	63.53	67.84	72.41	72.95
13	66.11	67.49	72.50	72.71
14	63.39	70.40	72.95	73.1
15	70.17	69.55	72.88	73.13
16	68.38	67.97	72.08	72.64
17	69.59	67.48	70.93	71.43
18	68.97	67.44	71.57	71.4
19	67.73	67.67	71.04	70.86
20	66.22	68.13	71.79	71
21	70.71	67.63	71.67	72.01
22	64.69	68.54	72.75	72.93
23	71.61	68.75	73.00	72.9
24	66.85	67.51	72.09	72.56
25	69.54	68.84	71.98	72.28
26	63.97	67.90	71.86	72.73

Number	Percentage of force degradation (%)			
	24 hours	1 week	2 weeks	4 weeks
27	64.68	67.32	72.29	72.79
28	60.55	68.13	71.74	72.68
29	60.95	69.06	71.21	71.79
30	64.47	69.10	73.23	74.02

8. Data of percentage of force degradation of latex elastic ligatures in force degradation experiment at 24 hours, 1 week, 2 weeks and 4 weeks.

Number	Percentage of force degradation (%)			
	24 hours	1 week	2 weeks	4 weeks
1	12.41	14.03	25.83	29.16
2	14.29	22.79	28.59	28.19
3	17.14	20.20	25.29	28.86
4	17.89	26.68	28.06	31.24
5	14.39	21.39	24.22	25.12
6	15.23	25.43	31.52	30.31
7	16.29	19.39	28.53	29.69
8	16.87	25.35	25.43	28.59
9	16.51	24.04	24.98	28.62
10	18.04	22.69	26.13	30.37
11	19.57	23.35	28.84	28.73
12	15.46	24.95	25.16	29.3
13	16.19	18.06	26.49	24.08
14	15.43	22.74	24.92	27.5
15	18.41	25.50	25.76	30.92
16	14.82	20.27	27.92	26.38
17	15.24	22.83	23.32	23.6
18	15.70	21.98	25.68	26.64
19	18.61	24.08	27.80	29.51
20	13.12	22.32	27.03	26.56
21	16.53	25.84	25.46	25.8
22	18.55	24.70	25.00	29.15
23	17.69	27.37	28.65	30.55
24	12.20	19.97	24.16	24.23
25	17.89	26.68	28.06	31.24
26	17.08	27.35	26.70	28.67

Number	Percentage of force degradation (%)			
	24 hours	1 week	2 weeks	4 weeks
27	16.50	22.52	28.15	27.82
28	17.40	23.22	28.04	28.54
29	17.69	27.37	28.65	30.55
30	15.97	27.37	24.86	27.99

9. Data of final dimensions of elastomeric ligatures (Unitek®) in permanent deformation experiment.

Number	Dimension (mm.)		
	Wall thickness	Outside diameter	Inside diameter
1	.57	4.48	3.35
2	.59	4.58	3.41
3	.55	4.49	3.40
4	.63	4.56	3.31
5	.63	4.54	3.29
6	.56	4.45	3.33
7	.58	4.47	3.31
8	.60	4.49	3.29
9	.58	4.49	3.33
10	.59	4.46	3.28
11	.57	4.50	3.37
12	.57	4.50	3.37
13	.59	4.51	3.34
14	.58	4.53	3.37
15	.56	4.47	3.35
16	.50	4.37	3.37
17	.57	4.56	3.42
18	.58	4.45	3.29
19	.58	4.43	3.28
20	.62	4.58	3.35
21	.61	4.57	3.35
22	.61	4.50	3.29
23	.61	4.56	3.35
24	.59	4.48	3.30
25	.61	4.53	3.32
26	.62	4.58	3.34

Number	Dimension (mm.)		
	Wall thickness	Outside diameter	Inside diameter
27	.59	4.52	3.34
28	.57	4.48	3.35
29	.69	4.56	3.18
30	.60	4.63	3.44

10. Data of final dimensions of latex elastic ligatures in permanent deformation experiment.

Number	Dimension (mm.)		
	Wall thickness	Outside diameter	Inside diameter
1	1.10	3.66	1.47
2	1.06	3.58	1.47
3	1.03	3.64	1.59
4	1.11	3.67	1.46
5	1.07	3.66	1.53
6	1.11	3.65	1.44
7	1.02	3.58	1.55
8	1.09	3.68	1.50
9	1.05	3.68	1.58
10	1.05	3.64	1.54
11	1.05	3.66	1.56
12	1.05	3.64	1.55
13	1.04	3.65	1.57
14	1.03	3.59	1.54
15	1.00	3.55	1.55
16	1.02	3.55	1.52
17	1.08	3.64	1.49
18	1.05	3.64	1.55
19	1.02	3.54	1.51
20	.96	3.43	1.52
21	1.08	3.66	1.50
22	1.03	3.60	1.55
23	1.02	3.58	1.54
24	1.05	3.63	1.53
25	1.02	3.58	1.54
26	1.04	3.56	1.49

Number	Dimension (mm.)		
	Wall thickness	Outside diameter	Inside diameter
27	1.00	3.46	1.47
28	1.03	3.61	1.55
29	.94	3.43	1.55
30	1.03	3.59	1.54

11. Data of percentage of permanent deformation of elastomeric ligatures (Unitek®) in permanent deformation experiment.

Number	Percentage of permanent deformation (%)		
	Wall thickness	Outside diameter	Inside diameter
1	-46.95	37.42	196.46
2	-42.65	42.24	188.98
3	-47.34	39.01	193.1
4	-40.48	41.61	195.54
5	-40.76	40.12	191.15
6	-47.17	37.35	197.32
7	-44.23	37.54	182.91
8	-42.58	38.15	183.62
9	-44.23	38.15	184.62
10	-43.27	37.65	182.76
11	-45.67	38.89	190.52
12	-46.45	39.32	200.89
13	-44.81	38.34	192.98
14	-44.23	40.68	195.61
15	-45.89	38.39	188.79
16	-52.15	35.29	195.61
17	-46.48	38.6	194.83
18	-44.23	37.35	183.62
19	-44.98	37.15	187.72
20	-41.71	41.36	196.46
21	-42.99	39.33	193.86
22	-42.38	39.32	191.15
23	-43.19	40.31	199.11
24	-43.54	38.7	189.47
25	-43.46	38.53	193.81
26	-41.51	40.49	192.98

Number	Percentage of permanent deformation (%)		
	Wall thickness	Outside diameter	Inside diameter
27	-45.87	36.97	198.21
28	-45.93	38.27	191.3
29	-35.21	39.88	181.42
30	-42.79	40.73	184.3

12. Data of percentage of permanent deformation of latex elastic ligatures in permanent deformation experiment.

Number	Percentage of permanent deformation (%)		
	Wall thickness	Outside diameter	Inside diameter
1	4.78	9.58	17.6
2	-2.31	6.23	21.49
3	-6.39	5.51	26.19
4	0.45	7.62	20.66
5	3.9	9.58	18.6
6	8.87	11.96	17.07
7	1	9.82	24
8	-0.91	8.24	25
9	-3.67	7.92	28.46
10	0	9.31	25.2
11	-4.98	7.65	31.09
12	-5.86	5.2	25
13	-5.45	7.35	30.83
14	-3.76	7.81	28.33
15	-9.91	3.8	29.17
16	-0.49	5.97	16.03
17	-2.27	6.74	23.14
18	0.48	10.64	28.1
19	-5.14	4.12	19.84
20	-7.73	4.26	24.59
21	-0.46	8.28	23.97
22	-1.91	9.42	29.17
23	-0.97	7.83	22.22
24	-5.41	6.14	27.5
25	-6.85	5.29	27.27
26	1.47	6.91	15.5

Number	Percentage of permanent deformation (%)		
	Wall thickness	Outside diameter	Inside diameter
27	-8.72	2.37	22.5
28	-7.21	5.87	30.25
29	-10.9	1.78	23.02
30	-6.39	5.9	28.33

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