

EFFECT OF VARIOUS CURING CONDITIONS IN PRESSURE COOKER ON FLEXURAL
STRENGTH OF DENTURE HARD RELINING MATERIALS



A Thesis Submitted in Partial Fulfillment of the Requirements
for the Degree of Master of Science in Prosthodontics

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ชนิดแข็ง



วิทยานิพนธ์นี้เป็นส่วนหนึ่งของการศึกษาตามหลักสูตรปริญญาวิทยาศาสตรมหาบัณฑิต

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Field of Study Prosthodontics
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งานวิจัยนี้ เป็นการประเมินผลของการบ่มตัวในหม้ออัดความดันในสภาวะต่างๆ ต่อความแข็งแรง
ดัดโค้งของวัสดุเสริมฐานฟันเทียมชนิดแข็ง โดยเตรียมชิ้นงานวัสดุเสริมฐานฟันเทียมชนิดแข็ง (Unifast™
Trad, Tokuyama® Rebase II Fast แซ่ Hardener® และ ไม่แซ่ Hardener®) เตรียมชิ้นงานสำหรับวัสดุแต่ละ
ชนิดจำนวน 70 ชิ้นและแบ่งออกเป็น 7 กลุ่ม ประกอบด้วยกลุ่มควบคุม และ 6 กลุ่มทดลองที่บ่มด้วยความดัน
อากาศหรือไนโตรเจนในหม้ออัดความดัน 2 บาร์ 55 องศาเซลเซียส เป็นเวลา 10, 15 และ 20 นาที ตามลำดับ
สำหรับกลุ่ม Tokuyama® Rebase II Fast ที่แซ่ Hardener® นำชิ้นงานแซ่ในสารละลาย Hardener® เวลา 3
นาทีภายหลังกการบ่มตัว ทดสอบความแข็งแรงดัดโค้งของชิ้นงานด้วยด้วยเครื่องทดสอบแรงดึงแรงอัด
วิเคราะห์ข้อมูลทางสถิติด้วยการวิเคราะห์ความแปรปรวนสามทางและทางเดียว เปรียบเทียบระหว่างกลุ่ม
ด้วยทุคีย์ (Tukey's HSD) ที่ระดับนัยสำคัญทางสถิติร้อยละ 95 ผลการวิเคราะห์ความแปรปรวนสามทาง
พบว่า ชนิดวัสดุและสภาพแวดล้อมมีผลต่อความแข็งแรงดัดโค้ง ($P < 0.05$) แต่เวลาบ่มตัวไม่มีผลต่อความ
แข็งแรงดัดโค้ง ($P > 0.05$) ในวัสดุชนิดเดียวกันกลุ่มที่อัดด้วยอากาศและไนโตรเจนมีความแข็งแรงดัดโค้งสูง
กว่ากลุ่มควบคุมอย่างมีนัยสำคัญ ($P < 0.05$) โดยวัสดุกลุ่มที่บ่มด้วยไนโตรเจนมีความแข็งแรงดัดโค้งสูงกว่า
กลุ่มที่อัดด้วยอากาศอย่างมีนัยสำคัญ ($P < 0.05$) ด้วยเงื่อนไขการบ่มเดียวกัน กลุ่ม Unifast™ Trad มีความ
แข็งแรงดัดโค้งสูงกว่ากลุ่ม Tokuyama® Rebase II Fast ที่แซ่และไม่แซ่ Hardener® อย่างมีนัยสำคัญ
($P < 0.05$) อย่างไรก็ตาม ความแข็งแรงดัดโค้งระหว่างกลุ่ม Tokuyama® Rebase II Fast ที่แซ่และไม่แซ่
Hardener® มีค่าไม่แตกต่างกันอย่างมีนัยสำคัญ ($P > 0.05$) ดังนั้นการบ่มในหม้ออัดความดันช่วยเพิ่มความ
แข็งแรงดัดโค้งของวัสดุเสริมฐานฟันเทียมชนิดแข็งอย่างมีนัยสำคัญ โดยการบ่มด้วยความดันไนโตรเจนด้วย
เวลาการบ่มที่เหมาะสมจะช่วยเพิ่มความแข็งแรงดัดโค้งอย่างมีนัยสำคัญมากกว่าการใช้ความดันอากาศ

สาขาวิชา ทันตกรรมประดิษฐ์

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EFFECT OF VARIOUS CURING CONDITIONS IN PRESSURE COOKER ON FLEXURAL STRENGTH OF DENTURE HARD RELINING MATERIALS. Advisor: Assoc. Prof. Chairat Wiwatwarrapan

This study evaluated the effect of various curing conditions in pressure cooker on flexural strength of hard chairside reline resins (Unifast™ Trad, Tokuyama® Rebase II Fast with and without Hardener®). For each material, 70 specimens were prepared and divided into 7 groups consists of control group and 6 experimental groups: cured in 2 bars air or nitrogen pressure cooker at 55°C for 10, 15 and 20 min respectively. For Hardener® groups, specimens were soaked in Hardener® for 3 min after curing. The flexural strength was tested using a Universal testing machine. The data were analyzed using Three-way ANOVA, One-way ANOVA and post hoc Tukey's HSD analysis at a 95 % confidence level. Three-way ANOVA result was showed that types of materials and curing environments affected on the flexural strength ($P < 0.05$) but curing time did not affect on the flexural strength ($P > 0.05$). In each material, the flexural strengths of air and nitrogen compressed groups were significantly higher than that of control group ($P < 0.05$). In each material, the nitrogen compressed groups also had significantly higher flexural strength compared with the air compressed groups ($P < 0.05$) With the same curing conditions, the Unifast™ Trad groups had significantly higher flexural strength than the Tokuyama® Rebase II Fast groups with and without Hardener® ($P < 0.05$). However, there was no significant difference in the flexural strength between the Tokuyama® Rebase II Fast groups with and without Hardener® ($P > 0.05$). Curing in the pressure cooker increased the flexural strength of the hard chairside reline resins. Moreover, using nitrogen gas pressure with satisfactory curing duration also increased the flexural strength compared with using air pressure.

Field of Study: Prosthodontics

Student's Signature

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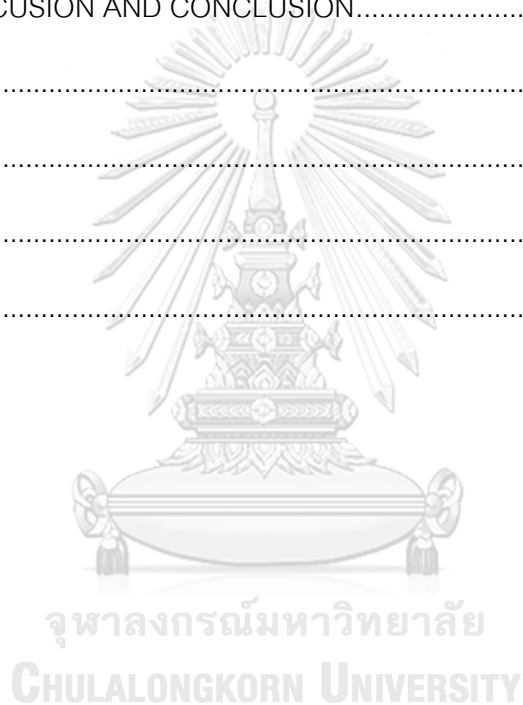
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CHAPTER 1

INTRODUCTION

Background and rotational

Acrylic based removable denture has been used to replace missing teeth continuously, due to poly(methyl methacrylate) (PMMA) or acrylic resin properties including non-toxicity, low cost, uncomplicated process ability and biocompatibility.[1-3] Even though, there are many advantages but the obstacles always arise because of the usual consequences.

After loss of teeth, the residual alveolar ridge goes through bone remodeling, including bone resorption and bone formation. Results change of bone configuration. This reduction of the residual alveolar ridge occurs most expeditiously since tooth extraction in first 6 months to 2 years [4], which lead to inadaptability of the former denture base and also reducing capacity of speech and mastication. For the resorbed residual alveolar ridge, the denture base is needed to be relined to provide comfort to patient by recovering the biomechanical properties and occlusion [5], improve adaptation to the residual ridge, restore fit, improve support retention and stability of denture base [6, 7]. Autopolymerizing acrylic resin can be used to directly reline in the mouth. This method is considered as less cost-consuming technique, easy to manipulate, not time-consuming and not keep the patient's denture.[6] In addition, autopolymerizing acrylic resin has shown satisfactory physical and mechanical properties. Autopolymerizing acrylic resin in is suitable for various situations including for maladapted prostheses poor retention and stability both at the delivery moment and after been used and give excellent outcomes.[5] One of the obviously disadvantages of autopolymerizing acrylic resin is unreacted methyl methacrylate (MMA) [8] or residual monomer which achieved by using the chemical activator and resulted the low degree of conversion.[9] Many researchers found that several autopolymerizing acrylic resin shown high percentage of residual monomers, which can compromise the physical properties, such as the less glass transition temperature [10], more flexibility of the material, less tensile strength, and increase water absorption [11]. Residual monomer

was found acting as a plasticizer, decreased flexural strength.[12] In addition, residual monomer also cause irritation, inflammation and allergic reaction of oral tissues.[13] For these reasons, various methods have been used to reduce the residual monomer and improved some mechanical properties. These methods are such as soaking the relined denture in hot water (50-55°C) [9], immersion in water for 24 hours after polymerization [14] and using the microwave post-polymerized treatment [15].

In addition, the disadvantages of relining with autopolymerizing acrylic resin can be modulated with using the curing environment in compressed air and water. This technique is known as the indirect technique for curing autopolymerizing acrylic resin. This improved the dimensional accuracy and was less occlusion changes during curing. The porosity of the material, cured with this technique, was less than that of the material cured at room pressure.[16] Some researches assumed that oxygen can inhibit or retard polymerization of acrylic resin either conventional or cross-linked material. In addition, curing autopolymerizing acrylic resin in water, in purpose to exclude air, was the key factor to reduce residual monomer and achieve adequate degree of polymerization on the surface of autopolymerizing acrylic resin. So, elimination of oxygen during polymerization might improve the mechanical properties.[17-19]

Therefore, this research is used a pressure cooker and replacing air with nitrogen gas, as purging gas. Various condition of curing time are the parameters to investigate the efficacy on the flexural properties of the MMA-based and non MMA-based hard chairside reline resins to verify if they are effective.

Research question

Do hard chairside reline resins, were cured in various condition in pressure cookers, increase the flexural properties of the reline materials?

Research Objective

To study the effect of various curing condition in pressure cookers to hard chairside reline resins in term of the flexural strength.

Hypothesis

Ho₁: The flexural strength of those hard chairside reline resins cured in pressure cooker is not statistically significant different from that of hard chairside reline resins cured in room atmosphere. (P value >0.05)

Ha₁: The flexural strength of those hard chairside reline resins cured in pressure cooker is statistically significant different from that of hard chairside reline resins cured in room atmosphere. (P value <0.05)

Ho₂: The flexural strength of those hard chairside reline resins cured in nitrogen pressure cooker is not statistically significant different from that of hard chairside reline resins cured in air pressure cooker. (P value >0.05)

Ha₂: The flexural strength of those hard chairside reline resins cured in nitrogen pressure cooker is statistically significant different from that of hard chairside reline resins cured in air pressure cooker. (P value <0.05)

Ho₃: The various curing times (10, 15, 20 mins) do not statistically significant affect on the flexural strength of those hard chairside reline resins cured in pressure cooker. (P value >0.05)

Ha₃: The various curing times (10, 15, 20 mins) statistically significant affect on the flexural strength of those hard chairside reline resins cured in pressure cooker. (P value <0.05)

Ho₄: The flexural strength of various hard chairside reline resins with the same cured condition are not statistically significant different. (P value >0.05)

Ha₄: The flexural strength of various hard chairside reline resins with the same cured condition are statistically significant different. (P value <0.05)

Ho₅: Tokuyama[®] Resin Hardener do not statistically significant affect on the flexural strength. (P value >0.05)

Ha₅: Tokuyama[®] Resin Hardener statistically significant affect on the flexural strength. (P value <0.05)

CHAPTER 2

REVIEW LITERATURE

Alveolar resorption

After tooth extraction, the residual alveolar ridge undergoes bone remodeling, including bone resorption and bone formation. This change of bone configuration occurs rapidly in first 6 months to 2 years after tooth extraction both on maxillary and mandibular. The loss of bone happened continuously even without a denture on the residual alveolar ridge.[4, 20, 21] Ridge form alteration is especially unavoidable in denture wearers, whose bone loss in a row over the years because of occlusal forces on the gingival tissues irritate bone. These results diminish in bone volume and density.[22] Finally, this will lead to inadaptability of denture base and may causes lessen of stability of denture base in patient's mouth. For those reasons, denture should be follow up periodically to detect and recover the problem instantly.[23]

Denture base polymer

According to ISO 20795-1:2013, denture base polymers were classified as the following type and classes[24]

Type 1: Heat-polymerizable materials

- Class 1: Powder and liquid
- Class 2: Plastic cake

Type 2: Autopolymerizable Materials

- Class 1: Powder and liquid
- Class 2: Powder and liquid for pour-type resins

Type 3: Thermoplastic blank or powder

Type 4: Light-activated materials

Type 5: Microwave cured materials

Autopolymerizing acrylic resin was used as the repair or relined material of denture base. Instead of using heat as an initiator, autopolymerizing acrylic resin was

added 3° aromatic amine into liquid compartment to allow autopolymerizing. 3° aromatic amine is an activator or accelerator that cause decomposition of benzoyl peroxide, which act as initiator, and become free radicals. The oxidation of 3° aromatic amine can cause color instability. However, addition of stabilizing agent can prevent oxidation.[1]

Table 1 Principle ingredient of acrylic resin in autopolymerizing acrylic resin [25]

	Ingredient	Chemical composition
Powder	Acrylic polymer	Polymethyl methacrylate
	Initiator	Benzoyl peroxide, Diisobutylazonitrile
	Pigment	Mercuric sulfide, Ferric oxide
	Dyes	Tissue or tooth like color
	Opacifier	Zinc or titanium oxide
	Plasticizer	Dibutyl phthalate
	Dyed organic fibers	Nylon, Acrylic
	Inorganic particles	Glass fiber, Zirconium silicate
Liquid	Monomer	Methyl methacrylate
	Inhibitor	Hydroquinone
	Activator or accelerator	<i>N, N</i> -dimethyl-para-toluidine or <i>N, N</i> -dihydroxyethyl-para-toluidine
	Plasticizer	Butyl or Octyl methacrylate
	Crosslink agent	Glycol dimethacrylate

Polymer- monomer interaction [1, 26]

When the powder and liquid are mixed, a series of physical changes take place before polymerization. After the powder is mixed into the liquid, the mixture is somewhat “sandy”. There is no or little interaction in molecular level. The polymer bead stays contacted. The consistency is course or grainy. Then, the monomer starts to diffuse into the polymer bead, and swell them. Some polymer molecular chains distribute in liquid monomer. The mixture become sticky and fibrous. So, it is often called “stringy stage”. Within a minute the material become less glossy and no longer stick to the finger. More polymer chains enter the monomer solution. The undissolved polymer bead suspended

in the matrix of monomer and dissolved polymer. The mixture become “*dough-like*” and appropriate to pack into the mold.

As monomer continues to penetrate completely into the center of polymer bead. The mixture is “*rubber-like*” and can rebound when stretched or compressed. Autopolymerizing acrylic resin begins the polymerization at this point and harden within minutes.

Upon an extended period, the mixture become “*stiff*”. This can be attributed to continuous monomer evaporation. From clinical viewpoint, the mixture is dry and resisted to deformation.

Polymerization mechanism [1, 27]

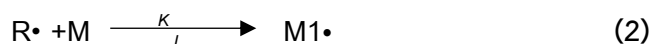
Polymerization reaction of poly(methyl methacrylate) is a radical chain or addition polymerization. This polymerization consisting of a sequence of three steps: initiation, propagation and termination.

The initiation step is considered to deal with two reactions. The first part is production of free radical or activation stage. The typical case is homolytic dissociation of an initiator (I) to a pair of radical (R^\bullet). Free radical can be formed by activation of an initiator using chemical agent, heat and visible light. For activation of a benzoyl peroxide initiator, the bond between the two-oxygen atoms is break down and the electron pair is separated between the two fragments. The two unpaired electrons appear on each side of the two free radicals



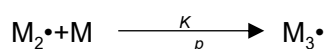
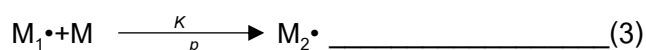
where the K_d is rate constant for the catalyst dissociation. One of the necessities of an addition polymerization is the possession of unsaturated group or double bond on reacting.

The second part is addition of this free radical to the first monomer molecule and alter the double bond $-C=C-$ into the single bond $-C-C-$. Thus, the originated free radical bond to monomer molecule and form a new free radical at the other side of the molecule. It is ready to react with another monomer molecule.

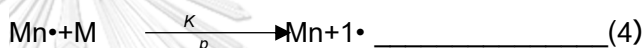


where the M represents a monomer molecule, $M_1\cdot$ is initial monomer radical and K_i is the rate constant for the initiation step.

The propagation step deals with the growth of $M_1\cdot$ by additions of huge amount of monomer molecules. Each addition results a new radical with the same identity as the one formerly. The additions are evinced by:



or in general terms



where K_p is the rate constant for propagation. Propagation with growth of the chain occurs very rapidly.

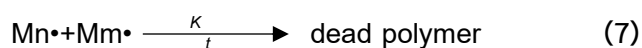
The termination step occurs by either combination of coupling or disproportionation. Combination of coupling takes place by mean of direct coupling of active centers from the two-propagating polymer. One growing chain confront another growing chain then both molecules combine and become deactivated by building a covalent bond.



Disproportionation occurs by mean of transferring a hydrogen atom from one growing chain to another. This cause a double bond to be formed when hydrogen atom is transferred. The two different modes of termination can be evinced by:



where K_{tc} and K_{td} are the rate constants for termination by coupling and disproportionation, respectively. It can be expressed the termination step by



where the particular mode of termination is not specified and $K_t = K_{tc} + K_{td}$.

Rate Expression

Monomer disappears by the initiation reaction as same as by the propagation reactions. The rate of monomer disappearance, which is equal to the polymerization rate, is given by

$$\frac{-d[M]}{dt} = R_i + R_p \quad (8)$$

where R_i and R_p are the initiation and propagation rates, respectively. However, the number of monomer molecules reacting in the initiation step is much less than those in the propagation step. So, R_i can be abandoned and the polymerization rate is given by the propagation rate

$$\frac{-d[M]}{dt} = R_p \quad (9)$$

The propagation rate, and also the rate of polymerization, is the sum of many single propagation steps. It is assumed that the rate constants of all the propagation steps are the same, so, it can be evinced the polymerization rate by

$$R_p = K_p [M\cdot] [M] \quad (10)$$

where $[M]$ is the monomer concentration and $[M\cdot]$ is the total concentration of all chain radicals, which included all radicals of size $M_1\cdot$ and larger.

The rate change of the radical concentration quickly alters and remains zero during the polymerization. It is assumed the rates of initiation (R_i) and termination (R_t) of radicals are equal or

$$R_i = R_t = 2K_t [M\cdot]^2 \quad (11)$$

The right side of the equation represents the rate of termination. The factor 2 in the termination rate equation is follows the convention for reactions breaking radicals in

pairs. It is also used for reactions that create radicals in pairs. Practically, the factor of 2 has not always been followed. So, the equation is rearranged to

$$[M\cdot] = \left(\frac{R_i}{2k_t}\right)^{1/2} \quad \text{_____} \quad (12)$$

and substitute $[M\cdot]$ (12) into the propagation rate (10) yields

$$R_p = k_p[M] \left(\frac{R_i}{2k_t}\right)^{1/2} \quad \text{_____} \quad (13)$$

Kinetics of Initiation and Polymerization

The rate of creating primary radicals by homolysis of an initiator (R_d) is given by

$$R_d = 2fk_d [I] \quad \text{_____} \quad (14)$$

where $[I]$ is the initiator concentration and f is the initiator efficiency. The initiator efficiency is considered as the fraction of the radicals obtained in the homolysis reaction that initiate polymer chains.

In initiation reaction, the second step (adding primary radical to monomer) is faster than the first step (production of free radical). The homolysis of the initiator is the rate-determining step of the initiation sequence, and the initiation rate is given by

$$R_i = 2fk_d [I] \quad \text{_____} \quad (15)$$

Substitute $[R_i]$ (15) into the rate of propagation equation (13) yields

$$R_p = k_p[M] \left(\frac{fk_d[I]}{k_t}\right)^{1/2} \quad \text{_____} \quad (16)$$

Dependence of Polymerization Rate on Initiator

The previous equation (16) shows that the polymerization rate is depended on the square root of the initiator concentration. However, the termination mode may convert from the bimolecular termination of two propagating radicals to primary termination, in which propagating radicals combine with primary radicals

$$R_i = k_d [\text{reductant}] [\text{oxidant}] \quad (19)$$

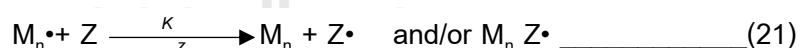
$$R_p = k_p [M] \left(\frac{k_d [\text{reductant}] [\text{oxidant}]}{2k_t} \right)^{1/2} \quad (20)$$

This equation is differed from initiation rate and propagation rate mentioned above. In this equation (19), the factor 2 is absent from the expression for R_i , because only one radical is obtained per oxidant-reductant pair.

Inhibition and retardation

The additional substances or impurity in the monomer can inhibit or retard polymerization. They react with the initiating and propagating radicals and reforming them into nonradical species or a too-low-reactivity radicals to undergo propagation. The polymerization suppressors are divided into two types according to their characteristic. *Inhibitors* stop every radical, and polymerization is completely stop. *Retarders* are less efficient and stop just a portion of the radicals. Thus, polymerization occurs, but in a slower rate. Substances that can Inhibits or retard spontaneous polymerization such as hydroquinone and a large amount of oxygen.

The kinetics of retardation or inhabitation can be delineate using the usual initiation, propagation, and termination reactions in addition to the inhibition reaction



where Z is the inhibitor or retarder. Z acts either by reacting to the propagating radical to form $M_n Z \cdot$ or by chain transferring of hydrogen atom to obtain $Z \cdot$ and polymer. This kinetics are assumed that $Z \cdot$ and $M_n Z \cdot$ do not initiate polymerization again and also terminate without Z regeneration. The assumption for the radical concentration leads to

$$\frac{d[M \cdot]}{dt} = R_i - 2k_t [M \cdot]^2 - k_z [Z] [M \cdot] = 0 \quad (22)$$

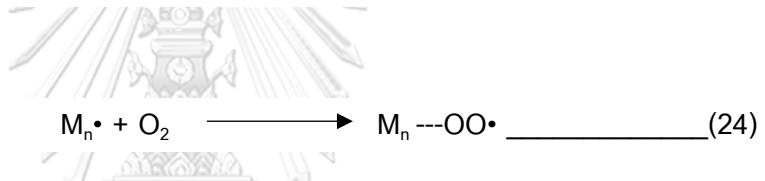
which can be combine with the rate of propagation equation to yield

$$\frac{2R_p^2 k_t}{k_p^2 [M]^2} + \frac{R_p [Z] k_z}{k_p [M]} - R_i = 0 \quad \text{_____} (23)$$

This equation has been used to correlate rate data in inhibited polymerizations. It shows that R_p is inversely proportional to K_z/K_p , the ratio of the rate constants for inhibition and propagation. The ratio is known as the inhibition constant z :

$$z = \frac{k_z}{k_p}$$

Oxygen is considered as a powerful inhibitor due to its huge z values (33,000 for of MMA polymerization). Oxygen reacts with radicals to form unreactive peroxy radical who reacts with itself or another propagating radical by means of coupling or disproportionation reactions to establish inactive products such as peroxides and hydroperoxides.



Peroxy radicals can also combine with monomer to obtain an alternating copolymer. The oxygen action is known as to initiate some polymerizations. Some commercial processes for ethylene polymerization interlace with oxygen initiation. With the reasons of that initiation can occurs by thermal decomposition of peroxides and hydroperoxides which formed from monomer or other impurities. Even though oxygen is an inhibitor or, unusually, an initiator will be related with highly temperature. Initiation will happen at higher temperatures where the peroxides and hydroperoxides are insecure.

Relining denture base

When clinical assessment on denture was found that adaptation to the supporting tissues is poor, the denture should be relined. However, the problems should be correctly diagnosed before considered relining as a treatment. Dentists should assess the clinical conditions of denture to ensure that the vertical dimension and the occlusal plane are correct, the centric occlusion and centric relation are harmony, the esthetics and the prosthetics teeth are acceptable and the teeth position is properly

related to the alveolar ridge.[28] There is contraindication of relining recommended in cases of prostheses with great tooth wear or malposition teeth; premature contacts or interferences; patients shown an inflamed or hyperplastic support mucosa; loss of vertical dimension more than 3 mm or lack of inter-occlusal space.[5] Besides, in newly fabricated distal extension removable partial denture, a small degree of error could happen during processing which is shrinkage of denture acrylic resin about 3-6% during polymerization.[29] In addition, there is a laboratory data shown that denture can be reached a better fitting by relining the heat polymerizing denture base with an autopolymerizing acrylic resin before placement.[30] In other words, one of the indication is poor adapted denture at the delivery moment and the check-up appointment.

Denture relining has been considered as a compromise treatment. There are two mainly methods of dentures relining: the indirect method or laboratory-process method, used heat polymerizing acrylic resins and the direct or chairside method, used autopolymerizing acrylic resins.[31] The indirect method requires making an impression, sending the prosthesis to a dental laboratory, and delivering it to patient, thus involving many patient visits, and also laboratory fee. The patients must be without dentures for a range of time. [16, 29, 31] This choice is characterized by the following disadvantages: reheating the denture could induce the warpage effect and releasing the residual internal stress.[16] However, the advantage of laboratory processing is a stronger, denser and more completely the cured reline resin compared with the direct reline materials.[29]

For the direct or chairside method, dentists directly relined denture with the autopolymerizing acrylic resins intraorally. This technique offers an immediate and inexpensive mean to the recondition.[29] So, the direct method is considered as a convenient method compared with the indirect method. Notwithstanding, this method has many deficiencies: it has unpleasant taste and odor to the patient; the flow of the material is hard to be controlled in patient's mouth; the appropriate occlusion is hard to maintain during the method; the interpolation is laborious if the position is wrong in the

first attempt; the border thickness and length are difficult to control; and also the tissue surface defects such as porous are common.[16]

To overcome those advantages of the indirect and direct method, an alternative procedure has been proposed.[32] The indirect relining method used autopolymerizing acrylic resin is a method to reline denture after making an impression. The master cast is made and the index material is used to replicate the tooth surface of denture on the lower part of the duplicated instrument. The master cast is mounted on the upper part of the duplicated instrument. After remove the impression material and place denture on the index material, mixed autopolymerizing acrylic resin is applied on the tissue surface. The duplicating instrument is closed completely and allowed the reline acrylic resin set under pressure of 2-2.5 bars for 30 minutes.[33] There is a study evaluate this technique using reline jig and allow autopolymerizing acrylic resin cure under water of at 37°C with pressure of 1.37 bars for 10 minutes. They reported the advantages of this technique: it can be completed within an hour after the impression is made; the denture need not be flaked so the separation is ease ; the tissue surface of the relined denture is less defects and porosity; the dimensional is accurate; and the changes in occlusion due to the processing are minimal.[16]

Hard chairside reline resin

It has been demonstrated that autopolymerizing acrylic resins present less flexural resistance when compared with thermal polymerizing acrylic resins, which can compromise the prosthesis after the relining procedure. These differences of mechanical properties may occur because of the higher amount of residual monomer after auto polymerization process when compared with the thermo-activated polymerization.[14, 34] The concerns also related to the direct reline materials were included porosity after polymerization which may lead to accumulate of microbial plaque in the long-term use.[35] Raising high temperature in some materials during polymerization could burn oral mucosa.[35] There is an unpleasant odor and taste.[35, 36] Some materials have more immediate dimensional change after polymerization

compared with the laboratory-processed material.[37] In addition, the residual monomer from uncompleted polymerization and leachable substance not only might stimulate allergic irritation and inflammation of oral mucosa, but also cause cytotoxic effect.[38-40]

The hard chairside reline resins can be divided into 2 groups by main composition of liquid constituents: MMA-based and non MMA-based reline materials. The MMA-based materials are available on the market such as Unifast™ Trad (GC Corp., Tokyo, Japan), Probase Cold® (Ivoclar, Liechtenstein) and Palapress Vario® (Heraeus Kulzer, Wehrheim, Germany). The MMA-based reline materials had better adhesion to the denture base due to MMA monomer that dissolve and penetrate into the denture base forming the interpenetrating polymer networks (IPN) bonds two layers of materials.[41, 42] They also had more flexural strength than the non MMA-based reline materials.[43] Although those advantages, the MMA-based hard reline resins have the problem of irritation to oral mucosa due to MMA irritation [44] or heat during polymerization. These problems have been resolved with the launch of the non MMA-based hard reline resins. The non MMA-based materials are available on the market such as Kooliner® (Coe Laboratories, Chicago, USA), Ufi gel hard® (Voco, Cuxhaven, Germany) and Tokuyama® Rebase II (Tokuyama Dental Corp, Tsukuba, Japan). The non MMA-based materials contain high molecular weight methacrylate. They are developed to help lessen irritation to the tissue.[45] In addition, they contain cross-linking agents in their liquid constituents, which improve the transverse strength.[46]

Table 2 composition of the MMA-based hard reline material [47]

Powder	Liquid
Powder: PMMA	Monomer: MMA
Initiator: benzoyl peroxide	Plasticizer: di-n- butylphthalate
	Chemical activator: tertiary amine

Table 3 composition of the non MMA-based hard reline material [47]

Powder	Liquid
Powder: PEMA	Monomer: butyl methacrylate or isobutyl
Initiator: benzoyl peroxide	methacrylate or other high molecular weight methacrylate monomer
	Crosslink agent: di-methacrylate
	Chemical activator: tertiary amine

Resin Hardener

A study reported that the polymerizing of cross-linked reline resins is interrupted by higher powder-to-liquid ratio, lower curing temperature and higher oxygen concentration. They also suggested that the oxygen-inhibited layer was easily stained and influences surface roughness.[17]

There is a study reported the mechanical of resin hardener of a hard reline material (Reverse[®] ((Nissin Dental Products Incorporated, Kyoto, Japan). The reducing agent (hardener) is consist of 1 wt.% sodium sulphite. They reported that the flexural strength was improved after immersed in 1 wt.% sodium sulphite solution. Furthermore, they assumed that the resin hardener help removed oxygen from the free radical exhibited on oxygen-inhibited layer to allow further polymerization, thus improve surface hardness and final polishing.[48]

For Tokuyama[®] rebase II, the resin hardener consists with two main ingredients: sodium bicarbonate 60-80% and sodium sulphite 20-40%. The resin hardener is prepared in hot water (40-60 °C) and polymerized material is immersed for 3 minutes. This can help complete the polymerizing top layer and avoid rough surface that influence bacteria growth and bad odor.

Residual Monomer

Autopolymerizing acrylic resin was shown higher residual monomer than heat polymerizing acrylic resin.[18, 34] It is because of lower degree of conversion of the autopolymerizing acrylic resin.[49, 50] Varying amounts of unreacted monomer or residual monomer remain within the polymerized resin.[15] Although pharmacological effect of residual monomers was not clear yet, there are many reports about health problems related with it. Many researches have a consistent observation that residual monomers effect on the oral soft tissue, circulatory system, digestive system, and respiratory system. In addition, cytotoxicity effect has been reported.[8, 38-40]

Residual monomer can affect on the physical properties such as the reduced the glass transition temperature [10], the decreased flexural strength of the material [12, 51], and also more allergic reaction [13], irritation and inflammation of oral tissue [5, 8]. Because of these reasons, various methods have been used to decrease the residual monomer, such as relined denture immersion in hot water (50-55°C) [9], immersion in water for 24 hours after completed polymerization [14], microwave post-polymerized radiation [15] and ultrasonic immersion in water [52-54] or ethanol solution [55, 56].

Curing time and temperature

Since the residual monomer can affect on the flexural strength. Increasing the degree of conversion of autopolymerizing acrylic resins could decrease the residual monomer and improve the flexural strength of cured materials.

Post-polymerization treatment for the cured material with high temperature environment had been recommended. Urban et al. reported that water bath post polymerization at 55°C for 10 minutes reduced the residual monomer of some hard chairside reline resin (Kooliner[®], New Truliner[®], Ufi gel hard[®] and Tokuso Rebase[®] Fast), increased degree of conversion of Kooliner[®] and Tokuso Rebase[®] Fast and also increased the flexural strength of Kooliner[®] from 32.5 MPa to 46.2 MPa.[9]

Urban et al. evaluated the effect of post polymerization treatment with microwave irradiation and water bath on the residual monomer of some commercial hard chairside

reline resin (Duraliner II, Kooliner[®], Tokuso Rebase[®] Fast and Ufi gel hard[®]) and one heat polymerizing acrylic resin (Lucitone500). They reported that immersion in hot water (55°C) for 10 minutes significantly reduced the residual monomer of hard chairside reline resins and a heat polymerizing acrylic resin.[15]

Rosangela et al. evaluated the effect of post-polymerization treatment by water-bath (55°C for 10 minutes) and microwave irradiation on flexural strength. They reported that microwave irradiation post-polymerization treatment increased the flexural strength of a heat polymerizing acrylic resin (Lucitone 550-L), while the other increased the flexural strength of the heat polymerizing acrylic resin and a hard chairside reline resin (Kooliner[®]). The flexural strength of Kooliner[®] was increased from 32.52 MPa to 46.17 MPa. It was note that the flexural strength of the reline materials (Duraliner II, Ufi gel hard[®] and Tokuso Rebase[®] Fast) were not affected by any of the post-polymerization treatments.[12]

Vallittu et al. reported that increasing the curing temperature for autopolymerizing acrylic resins from 30°C to 60°C and curing time 15 minutes decreased the residual MMA content of the polymer from an average of 4.6 wt% to 3.3 wt%.[34]

Lee et al. evaluated effect of the three curing factors: temperature (50°C), pressure (2.5 bars) and curing environment (water or air) on the monomer elution and microhardness of autopolymerizing acrylic resins. The specimens were prepared by method that let the top surface exposed to curing environment. The specimen was left to polymerize undisturbed for 15 minutes. They reported that when curing in water with increased temperature, resin surface hardness was improved and the residual monomer was decreased, whether with or without pressure. It was summed that increasing temperature and curing in water significantly reduced the amount of residual monomer from autopolymerizing acrylic resins specimens. However, it was note that the pressure factor showed no significant influences on monomer elution and microhardness.[18]

Ogawa et al. evaluated the effects of the curing environment, air or water, and water temperature (10°C, 23°C, 30°C, 40°C, 60°C, and 80°C) during polymerization on

the flexural strength of an autopolymerizing acrylic resin (Unifast II). They reported that increasing temperature during polymerization showed a significant effect on both the flexural strength and modulus of the resin. Increasing water temperature up to 60°C and 80°C produce 2 times greater flexural strength of the resin compared with resin polymerized in 23°C air.[57]

Pressure cooker

Dental pressure vessel or pressure pot has been suggested with rationale of the pressurized environment which helps decrease volume loss associated with polymerization shrinkage, decrease air inclusions in powder and liquid mixture, promote flow of impression and replication materials and enhance surface characteristics.[58] In addition, curing autopolymerizing acrylic resin under pressure can lessen the porosity and prevent defect and void formation of the material.[16] Thus, autopolymerizing acrylic resin are typically cured in pressurized environment to enhance polymerization. Many studies have reported study of the relation of pressure cooker polymerization technique and mechanical properties including flexural strength of autopolymerizing acrylic resins.

Machado et al. studied effect of various curing conditions on mechanical properties of an autopolymerizing acrylic resin. It was found that curing in water at 50°C for 15 minutes under 1.37 bars can improve microhardness and impact strength of the autopolymerizing acrylic resin. They also stated that a better mechanical properties of autopolymerizing acrylic resin can be achieved with curing under controlled heat and pressure condition.[59]

Vallittu evaluated the effect of curing environment on thickness of the unpolymerized surface layer of autopolymerizing acrylic resin (Vario®). The specimens were polymerized at 55 °C either in water or in air under air pressure of 1 bar or 3 bars. Samples polymerized in water had no inhibition layer, while the samples polymerized in air had an inhibition layer of varying thickness. Increasing air pressure from an ambient

to 1 bar and 3 bars substantially increased the thickness of the unpolymerized surface layer.[19] Thus, there is a recommended that autopolymerizing acrylic resin should be cured in water under pressure to reduce the inhibition effect of oxygen on free radical polymerization.[25]

Donovan et al. determined the flexural strength, porosity, and hardness of autopolymerizing acrylic resin polymerized under various conditions: in ambient air, under water (21°C), under air pressure (1.37 bar), and under water (21°C) with air pressure (1.37 bar). They reported that autopolymerizing acrylic resin cured under 1.37 bar air pressure, whether with or without water resulted in higher flexural strength and less porous than that processed at ambient pressure, while curing specimens in water seems to have no additional effects on the variables tested.[60]

Curing atmosphere

It has been proved that oxygen prohibit polymerization and affect on the mechanical properties of acrylic resin.[19] It was also stated that the reactivity of oxygen and free radicals is higher than that of free radicals with monomers, thus, the polymerization reaction is inhibited by oxygen.[61]

Yatabe et al. evaluated properties resulting with unpolymerized surface layer on 6 hard chairside reline resins. The materials were polymerized in air at 24 and 37°C, and in distilled water at 37°C, thus, reduce oxygen exposure. The powder-to-liquid ratio was changed by ± 20 wt% of the manufacturer's recommends. They reported that especially on cross-linked reline materials, the unpolymerized layer was reduced with higher temperature, lower powder-liquid ratio, and lower oxygen existence.[17]

Lamb et al. evaluated the effect of different curing environment on level of residual monomer in an autopolymerizing acrylic resin. The specimens were cured at 3.5 bar at 22°C or 55°C. Some samples were rapidly opened to the atmosphere. The others were left enclosed to eliminate air exposure. All specimens were store in water temperature of 22°C, 37°C or 50°C. They found that lowest amount of residual monomer was achieved only in those samples from which air was excluded. It was stated that the

role of oxygen in free radical-addition polymerization is often to inhibit polymerization. Further polymerization of residual monomer was also inhibited when cured resin was exposed to air rapidly.[62]

However, a comparative study of the effect of nitrogen gas in pressure cooker curing method on the flexural strength of hard chairside relining resins has not yet been reported.

Flexural strength

The flexural strength of denture base and chairside relining resins are an important mechanical property. Many factors such as residual monomer, relining technique and method, temperature and pressure during cure process and post-polymerization treatments also affect on the properties. According to recommended by International Organization for Standardization, the measurement of the flexural strength is often taken by 3-point bending test.[24]

When determined in accordance with ISO 20795-1:2013 [24] the ultimate flexural strength shall be not less than 65 MPa for Type 1, Type 3, Type 4 and Type 5 polymers and not less than 60 MPa for Type 2 polymers (Table 4).

Table 4 The required properties of denture base polymer materials per ISO 20795-1:2013

Flexural properties					
	Ultimate flexural strength	Flexural modulus	Residual MMA monomer	Sorption	Solubility
	MPa	MPa	%mg	$\mu\text{g}/\text{mm}^3$	$\mu\text{g}/\text{mm}^3$
	min	min	max	max	max
Types 1,3,4, and 5	65	2,000	2.2	32	1.6
Type 2	60	1,500	4.5	32	8.0

There are studies reported the flexural strength of reline acrylic resin. At thickness of 3.0 mm, the mean of flexural strength of ProBase Hot was 72 MPa while Unifast™ Trad and Tokuyama® Rebase II Fast showed the mean of flexural strength of 53.08 and 29.95 MPa respectively.[43] The other study compared the flexural strength of a heat polymerizing denture base polymer and hard chairside reline resins including Unifast™ Trad and Tokuyama® Rebase II Fast. At thickness of 3.0 mm, ProBase Hot, Unifast™ Trad and Tokuyama® Rebase II Fast showed the mean of flexural strength of 77.63, 52.63 and 35.24 MPa Respectively.[63]



CHAPTER 3 METHODOLOGY

1. Population

Denture Hard Relining Materials

2. Study population

Unifast™ Trad and Tokuyama® Rebase II Fast denture hard reline materials

3. Study sample

70 specimens prepared from Unifast™ Trad and 140 specimens prepared from Tokuyama® Rebase II Fast denture hard reline materials

Material

Hard chairside reline resins: Unifast™ Trad

Hard chairside reline resins: Tokuyama® Rebase II Fast



Figure 1 Materials used in the present study. (a) Unifast™ Trad, (b) Tokuyama® Rebase II Fast

Sample preparation

Two-hundreds and ten strip pattern specimens were made of hard chairside reline resin (Unifast™ Trad and Tokuyama® Rebase II Fast) (Figure 1). First 70 specimens (group1-7) were prepared from Unifast™ Trad in the stainless-steel mold (Figure 2) with dimension of 64x10x3.3 mm (Figure 3) following the manufacturing instruction (Table 5) with different seven curing conditions as described as follows. For control group, Unifast™ Trad powder and liquid was mixed and applied in the mold. The mold was covered with glass slap to get rid excessive material, and topped with 1 kg of iron. Specimen cured in room temperature ($25\pm 1^{\circ}\text{C}$) and atmospheric pressure until it

reaches setting time (Unifast™ Trad; 2mins and Tokuyama® Rebase II Fast; 6mins), then removed the specimen from the mold.

For air groups, the pressure cooker (Figure 4) (IMT; Pressurepotter 003, Inmotion technology limited, Thailand) was connected with air pump (Figure 5[a]). The pressure cooker was filled with water below the specimen level and the temperature control panel was adjusted to 55°C. The stainless-steel mold with the material was placed on the stand inside the pressure cooker and the lid was closed tightly, and compressed with air 2 bars. The specimens were cured for 10, 15 and 20 minutes, respectively. For the nitrogen groups, the pressure cooker was connected with a nitrogen tank via a polyurethane tube (Figure 5[b]). The tip of the polyurethane tube was placed under water level. After adjust temperature to 55°C, the nitrogen valve was opened to let nitrogen gas flow into the pressure cooker. The pressure release valve of the pressure cooker was opened simultaneously to purge the air with nitrogen gas for 5 second. The pressure release valve was closed to rise the pressure to 2 bars. The specimens were cured under nitrogen gas pressure for 10, 15 and 20 minutes respectively.

Table 5 Chemical composition and manufacturer of testing materials

Material	Major ingredients	Mixing time	Working time	Powder-liquid ratio	Setting time	Manufacturer
Unifast™ Trad	Powder: PMMA, MMA&EMA copolymer Liquid: MMA monomer, dimethyl-p-toluidine	10-15 sec	2 mins	1.0g / 0.5ml	2 mins	GC Corporation, Tokyo, Japan
Tokuyama® Rebase II Fast	Powder: PEMA Liquid :1,9-NDMA, AAEMA Hardener® : Sodium bicarbonate, Sodium sulphite	5-10 sec	20 -60 sec	2.40g / 1.0ml	6 mins	Tokuyama dental corporation, Tokyo, Japan

PMMA, Poly (methyl methacrylate); MMA&EMA copolymer, Methyl methacrylate & Ethyl methacrylate copolymer; PEMA, Poly (ethyl methacrylate); 1,9NDMA, 1,9nonanedioldimethacrylate; AAEMA, 2-(acetoacetoxy) ethylmethacrylate.

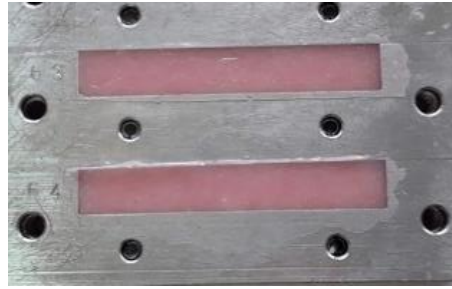


Figure 2 Stainless steel mold with loaded material

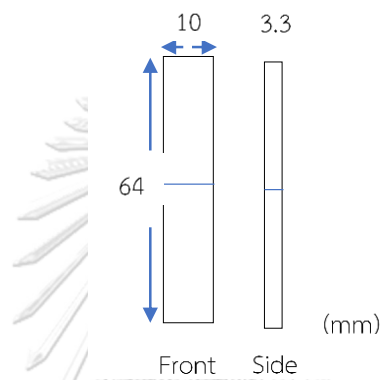


Figure 3 Illustration of the specimen strips size of 64x10x3.3 mm



Figure 4 Pressure cooker with pressure release valve on the top surface of the lid. The temperature control panel is on the front side of the set up.



Figure 5 (a) pressure cooker connected with air pump
(b) pressure cooker connected with nitrogen gas



Figure 6 (a) lay the flat surface symmetrically on the supports of the flexural test rig
(b) flexural strength test, using 3-point loading universal testing machine

Another 70 specimens (group8-14) were prepared from Tokuyama[®] Rebase II Fast using the same procedures as described for Unifast[™] Trad. For these specimens, after removed from the mold, were soaked in a Hardener[®] water solution (40-60°C) for 3 minutes then rinsed and dried. The other 70 specimens (group15-21) were also prepared from Tokuyama[®] Rebase II Fast but different in that these specimens were not soaked in Hardener[®] solution after removed from the stainless-steel mold.

The 210 specimens were distributed into 21 groups (n=10 for each group) followed their curing conditions as summarized in Table 6.

Table 6 Group of specimens

Group	Specimen	Atmosphere	Pressure cooker 55oC 2 Bar	number
1	Unifast™ Trad	Air	-	10
2		Air	10 min	10
3		Air	15 min	10
4		Air	20 min	10
5		Nitrogen	10 min	10
6		Nitrogen	15 min	10
7		Nitrogen	20 min	10
8	Tokuyama® Rebase II Fast (soak with Hardener®)	Air	-	10
9		Air	10 min	10
10		Air	15 min	10
11		Air	20 min	10
12		Nitrogen	10 min	10
13		Nitrogen	15 min	10
14		Nitrogen	20 min	10
15	Tokuyama® Rebase II Fast (no soak with Hardener®)	Air	-	10
16		Air	10 min	10
17		Air	15 min	10
18		Air	20 min	10
19		Nitrogen	10 min	10
20		Nitrogen	15 min	10
21	Nitrogen	20 min	10	

All specimens were polished with a metallographic grinding paper, roughly 30 micrometer (P500), on a polishing machine (NANA2000, Pace Technologies, USA) by wet grinding technique on both sides into thickness of 3.3 mm. All specimens were stored in distilled water within incubator (Contherm 160M Contherm Scientific Ltd., New Zealand) at a temperature of 37 ± 1 °C for 50 ± 2 hours prior to flexural testing.

Ultimate flexural strength

The flexural strength test was done following ISO 20795-1:2013.[24] Specimen strips were taken from water storage and immediately lay the flat surface symmetrically on the supports of the flexural test rig (SHIMADZU; EZ-S, SHIMADZU, JAPAN) (Figure 6). The force was increased on the loading plunger from zero, uniformly, using a constant displacement rate of 5 ± 1 mm/min and span of 50 mm and 500 N load cell until the specimen breaks. Flexural strength (σ , MPa) was calculated using the following equations:

$$\sigma = \frac{3Fl}{2bh^2}$$

F = maximum stress (N)

l = the distance between supports (mm)

b = mean of specimen width (mm)

h = mean of specimen height (mm)

Statistically analysis

The data were analyzed using SPSS for windows 22.0 (SPSS Inc., Chicago, IL, USA). The data of each groups were tested using Kolmogorov-Smirnov test to determine normal distribution of mean flexural strength. The equality of variance was tested using Levene's test. If the data shown a normal distribution ($P>0.05$) and homogeneous variance ($P>0.05$), three-way and one-way analysis of variance (post hoc Tukey HSD), 95 % confidence level would be used to analyze.

CHAPTER 4

RESULTS

Results

The data were analyzed using the Kolmogorov-Smirnov test to determine data distribution. The results shown that all data were normal distributed in all groups ($P>0.05$) (Table 12 in Appendix). The homogeneity of variance was determined using the Levene test. In case equal variances were assumed (Table 13-22 in Appendix)

3-way ANOVA result in Table 7-10 were showed that product and atmosphere affected on the flexural strength ($P<0.05$) but curing time did not affect on the flexural strength. There is interaction between product and atmosphere ($P<0.05$) but there is no interaction between product-time, atmosphere-time and product-atmosphere-time ($P>0.05$)

Table 7 P-values from 3-way ANOVA for effect of product, atmosphere, time and their interaction on the flexural strength

Variables	Flexural strength
Product	<0.001
Atmosphere	<0.001
Time	0.598
product x atmosphere	<0.001
product x time	1.000
atmosphere x time	.910
product x atmosphere x time	1.000

Table 8 The flexural strength values (mean (SD)) effect by product (MPa)

Product	Flexural strength values (mean (SD))
Unifast™ Trad (UT)	76.01(5.01) ^A
Tokuyama® Rebase II with Hardener® (TR+H)	49.82(1.95) ^B
Tokuyama® Rebase II without Hardener® (TR-H)	49.03(1.87) ^B

Table 9 The flexural strength values (mean (SD)) effect by atmosphere (MPa)

Atmosphere	Flexural strength values (mean (SD))
Air	56.52(12.02) ^A
Nitrogen gas	60.06(13.74) ^B

Table 10 The flexural strength values (mean (SD)) effect by time (MPa)

Time	Flexural strength values (mean (SD))
10 min	58.03(13.01) ^A
15 min	58.31(13.04) ^A
20 min	58.53(13.14) ^A

The mean flexural strength (MPa) and standard deviations of each group (n=10) are shown in Table 11. The result is divided as Unifast™ Trad (UT), Tokuyama® Rebase II Fast with Hardener® (TR+H) and Tokuyama® Rebase II Fast without Hardener® (TR-H) sequentially.

Table 11 The flexural strength values (mean (SD)) for each material at different curing conditions (MPa)

Curing conditions			Unifast™ Trad	Tokuyama®	Tokuyama®
pressure cooker (bar)	atmosphere	time (min)	(UT)	Rebase II with Hardener® (TR+H)	Rebase II without Hardener® (TR-H)
-	air	Control	64.88 (5.86) ^{A, a}	45.98 (1.52) ^{A, b}	44.58 (1.59) ^{A, b}
2	air	10	72.62 (3.22) ^{B, a}	48.35 (1.41) ^{B, b}	47.66 (0.83) ^{B, b}
2	air	15	73.16 (3.94) ^{BC, a}	48.80 (1.85) ^{BC, b}	48.03 (0.80) ^{BC, b}
2	air	20	73.17 (5.80) ^{BC, a}	48.74 (1.81) ^{BC, b}	48.14 (1.10) ^{BC, b}
2	N ₂	10	78.89(3.61) ^{CD, a}	50.69 (1.28) ^{CD, b}	49.94 (2.42) ^{CD, b}
2	N ₂	15	79.00 (3.50) ^{CD, a}	50.89 (1.76) ^{CD, b}	49.97 (1.69) ^{CD, b}
2	N ₂	20	79.24 (4.27) ^{D, a}	51.45 (1.29) ^{D, b}	50.43 (1.90) ^{D, b}

*** Same uppercase letter indicates no significant difference between the group in each column ($P>0.05$)

*** Same lowercase letter indicates no significant difference between the group in each row ($P>0.05$)

The data were analyzed using one-way ANOVA and Tukey HSD post hoc analysis to determine difference among them by each column (the same material, but various curing conditions) and each row (the various materials, but the same curing conditions).

The results of one-way ANOVA and post hoc analysis (Table 23-25 in Appendix), showed that the mean flexural strength of UT, TR+H and TR-H groups with curing under air and nitrogen gas pressure were significantly increased compared with that of the control group of the same material ($P < 0.05$).

In each hard reline material cured with the same environment in pressure cooker, there were no significantly different flexural strength of the groups with various curing time (10, 15, and 20 min) ($P > 0.05$). In addition, the flexural strength of groups cured under nitrogen pressure for 10 minutes were significantly higher than that of groups cured with air pressure for 10 min ($P < 0.05$). In contrast, there is no significantly different flexural strength between groups that cured under air pressure for 15 minutes and groups that cured under nitrogen gas pressure for 15 minutes ($P > 0.05$). However, the flexural strength those were cured under nitrogen gas pressure for 20 minutes, were significantly higher than groups those cured under air pressure for 20 minutes ($P < 0.05$).

With the same cured conditions, one-way ANOVA and post hoc analysis (Table 26-32 in Appendix) revealed that groups of UT showed significantly higher flexural strength than groups of TR+H and TR-H ($P < 0.05$). There was no significant difference on the flexural strength between groups of TR+H and TR-H. ($P > 0.05$).

CHAPTER 5

DISCUSSION AND CONCLUSION

Discussion

The hard chairside reline resins were classified as type 2 (autopolymerizing acrylic resin) class 1 (powder and liquid type) denture base polymer. Their polymerization was initiated with chemical reaction and did not requiring existence of curing temperatures above 65 °C to complete their polymerization.[24] The initiation reaction is redox reaction. The benzoyl peroxide initiator is activated by a reducing agent such as dimethyl-p-toluidine producing reactivated center (free radical). In this study, the flexural strength of hard chairside reline resins were investigated. Two brands of hard chairside reline resins, commercially available in Thailand, are Unifast™ Trad (MMA-based) and Tokuyama® rebase II Fast (non MMA-based). The disadvantages of hard chairside reline resins were lower flexural strength and more residual monomers due to less polymerization reaction. This study was determined that the effect of various curing condition in pressure cookers on the flexural strength of the reline materials.

In each hard reline material, the flexural strength of pressure cooker cured groups (55°C, 2 bar) were significantly higher than that of the control group (25°C, atmospheric pressure) because of the two factors. The first one is curing temperature, and the second is curing pressure. The curing temperature effects on the rate and degree of polymerization is important in determining the manner of performing polymerization. The increased curing temperature usually increases the polymerization rate and decrease the percentage of residual monomer.[27] The effect of pressure on polymerization is also important from the practical viewpoint since several monomers are polymerized at pressures above atmospheric. So that, high pressure can also have appreciable effects on increasing polymerization rates and polymer molecular weights.[27] Escalation of pressure had improved the flexural strength of the hard chairside reline resins, regardless of compressed with air or nitrogen gas. It is corresponding with previous studies reported that autopolymerizing acrylic resin cured

under pressure are shown lessen porous forming [60] and higher flexural strength [60, 64]. The pressure environment may prevent the monomer evaporation at the initial stage of polymerization, thus, minimized the porous formation within the material and improved flexural strength.[65] Based on these results, the first null hypothesis was rejected.

In addition, curing with nitrogen gas environment significantly increases the flexural strength of the hard relene materials because oxygen exposure was eliminated. There is a study reported the unpolymerized layer of hard chairside relene resin after cured autopolymerizing acrylic resin under air pressure. It was assumed that higher air pressure might provide more oxygen to resin surfaces and retard polymer chain growth and affect on the surface hardness.[19] Oxygen is a powerful inhibitor, as demonstrated by the very large inhibition constant values (ratio of the rate constants for inhibition and propagation). This value of MMA polymerization is 33,000.[27] Oxygen reacts with radicals to form the relatively unreactive peroxy radical that reacts with itself or another propagating radical by coupling and disproportionation reactions to form inactive products (probably peroxides and hydroperoxides). Consistent with a study reported that excluded air curing environment decreased residual monomer.[62] Radical-chain polymerization can be inhibited by oxygen which can reacts with free radicals. The large amounts of oxygen will compete for free radicals with MMA and inhibit polymerization. Oxygen has some characteristic like an unpaired electron, which can react with free radical initiator or during propagation of polymer chain. Thus, the degree of the inhibition is proportional to the concentration of oxygen.[25, 27, 65] However, the second null hypothesis was rejected.

For each hard relene material cured with the same environment in pressure cooker, there were no significantly different flexural strength of the groups with various curing time (10, 15, and 20 min). It may be explained by that degree of polymerization is terminated. The manufactures recommended that setting time of Unifast™ Trad is 2 minutes, while Tokuyama® rebase II Fast setting time is 6 minutes. So, increasing curing time to 10 15 and 20 minutes might not affect on the flexural strength. Thus, in this study assumed that curing under pressure, regardless compressed with air or nitrogen gas, at

2bars 55 °C for 10 minutes results the flexural strength equally to curing for 15 and 20 minutes. It is also note that manipulation follows the manufacturer's instructions results lower flexural strength. The third null hypothesis was accepted.

In the same curing conditions, the flexural strength of Unifast™ Trad is significantly higher than Tokuyama® rebase II Fast. This result is corresponding with previous studies.[43, 63] The difference in flexural strength of the two reline materials might be due to the differences of molecular structure and mechanical properties of the polymerized materials. The powder of Unifast™ Trad is PMMA while Tokuyama® rebase II Fast is mainly consisting of PEMA which the molecular size is larger than PMMA. The larger molecular size may increase space between the polymer chain and resulted lower flexural strength of the material. For Tokuyama® rebase II Fast, the liquid consists of 59% acetoacetoxy ethyl methacrylate (AAEM) monomer and 39% of 1,9-nonanediol dimethacrylate (1,9-NDMA) as the cross-linking agent.[66] The molecular weight of the monomer AAEM is 214.22 g/mol. The cross-linking agent 1,9-NDMA has a higher molecular weight of 296.4 g/mol.[66] Unifast™ Trad is mainly consist of MMA monomer and dimethyl-p-toluidine, and the molecular weight of MMA is 100 g/mol.[67] The higher molecular weight may be an obstacle for reacting with free radical, thus, resulted lower rate of polymerization and lower flexural strength of the material. In addition, because it is MMA-based, Unifast™ Trad has high exothermic behavior.[68]

During polymerization proceeds, the carbon-carbon double bonds (C=C) are converted to the carbon-carbon single bonds (C-C). The difference in energy between the two chemical bonds (80 kJ/mol) might emit as heat.[69] The emitted heat increased the curing temperature and polymerization reaction. This resulted higher flexural strength of Unifast™ Trad. The fourth null hypothesis was rejected.

For Tokuyama® rebase II Fast, there was no significant difference on the flexural strength between TR+H and TR-H. A study reported the effect of reducing agent of a commercial cross-linked reline material (Reverse® (Nissin Dental Products Incorporated, Kyoto, Japan)). They reported that reducing agent help removing oxygen from the free radical on the surface oxygen-inhibited layer of the reline material. Thus, allow the

polymerization to continue and the unpolymerized layer was further cured after immerse in reducing agent solution. The flexural strength of the cross-linked reline material was increase significantly after immerse for 15 minutes.[48] Although, the previous result is in contrast of the present study, it may be explained by that is due to the different method of the reducing agent immersion. In the present study, the upper surface of preparing specimen was sealed with glass slap in purpose to get rid of excessive matter. So, the specimen was seal from air during polymerizing. In addition, TR-H and TR+H were cured in pressure cooker at high temperature which already increase rate of polymerization and reduce residual monomer. The different flexural strength after application of Hardener[®] was not occur. The fifth null hypothesis was accepted.

Conclusion

Curing with pressure cooker significantly increased the flexural strength of the autopolymerized hard reline materials. In addition, curing in a pressure cooker with nitrogen atmosphere with appropriate curing time also significantly increase the flexural strength than curing with air atmosphere. So, Nitrogen pressure could be environment of choice to improved flexural strength of the hard reline materials.

REFERENCES

1. Anusavice KJ, Shen C, Rawls HR. Phillip's science of dental materials. 12th ed. Philadelphia: Saunders; 2003 p. 474-98.
2. Ali U, Karim KJ, Buang NA. A review of the properties and applications of poly (methyl methacrylate) (PMMA). *Polym Rev.* 2015;55(4):678-705.
3. Gautam R, Singh RD, Sharma VP, Siddhartha R, Chand P, Kumar R. Biocompatibility of polymethylmethacrylate resins used in dentistry. *J Biomed Mater Res B Appl Biomater* 2012;100B(5):1444-50.
4. Atwood DA. Bone loss of edentulous alveolar ridges. *J Periodontol.* 1979;50(4S):11-21.
5. Kubo CS, Amaral FR, Campos EA. Relining of removable dentures: a literature review. *RSBO.* 2014;11(2):192-8.
6. Leles CR, Machado AL, Vergani CE, Giampaolo ET, Pavarina AC. Bonding strength between a hard chairside relined resin and a denture base material as influenced by surface treatment. *J Oral Rehabil.* 2001;28(12):1153-7.
7. Takahashi Y, Kawaguchi M, Chai J. Flexural strength at the proportional limit of a denture base material relined with four different denture relined materials. *Int J Prosthodont.* 1997;10(6):508-12.
8. Krunic N, Kostic M, Najman S, Djindjic N. Toxicity testing of prosthetic metacrylates. *Healthmed.* 2012;6(7):2518-24.
9. Urban VM, Machado AL, Vergani CE, Giampaolo ET, Pavarina AC, Almeida FG, Cass QB. Effect of water-bath post-polymerization on the mechanical properties, degree of conversion, and leaching of residual compounds of hard chairside relined resins. *Dent Mater.* 2009;25(5):662-71.
10. Jerolimov V, Jagger RG, Milward PJ. Effect of the curing cycle on acrylic denture base glass transition temperatures. *J Dent.* 1991;19(4):245-8.
11. Dogan A, Bek B, Cevik NN, Usanmaz A. The effect of preparation conditions of acrylic denture base materials on the level of residual monomer, mechanical properties and

water absorption. *J Dent*. 1995;23(5):313-8.

12. Rosangela SS, Carlos EV, Eunice TG, Ana CP, Ana LM. Effect of post-polymerization treatments on the flexural strength and Vickers hardness of reline and acrylate denture base resins. *J Appl Oral Sci*. 2007 15(6):506–11.

13. Barclay SC, Forsyth A, Felix DH, Watson IB. Case report - hypersensitivity to denture materials. *Br Dent J*. 1999;187(7):350-2.

14. Vallittu PK, Miettinen V, Alakujala P. Residual monomer content and its release into water from denture base materials. *Dent Mater*. 1995;11(6):338-42.

15. Urban VM, Machado AL, Oliveira RV, Vergani CE, Pavarina AC, Cass QB. Residual monomer of reline acrylic resins: effect of water-bath and microwave post-polymerization treatments. *Dent Mater* 2007;23(3):363-8.

16. Smith DE, Lord JL, Bolender CL. Complete denture relines with autopolymerizing acrylic resin processed in water under air pressure. *J Prosthet Dent*. 1967;18(2):103-15.

17. Yatabe M, Yasuda N, Ai M, Okabe Y. Unpolymerized layer on autopolymerizing, hard reline materials. *Int J Prosthodont*. 1999;12(2):129-34.

18. Lee SY, Lai YL, Hsu TS. Influence of polymerization conditions on monomer elution and microhardness of autopolymerized polymethylmethacrylate resin. *Eur J Oral Sci*. 2002;110(2):179-83.

19. Vallittu PK. Unpolymerized surface layer of autopolymerizing polymethyl methacrylate resin. *J Oral Rehabil*. 1999;26(3):208-12.

20. Carlsson GE, Persson G. Morphologic changes of the mandible after extraction and wearing of dentures. A longitudinal, clinical, and x-ray cephalometric study covering 5 years. *Odontol Revy*. 1967;18(1):27-54.

21. Carlsson GE, Bergman B, Hedegard B. Changes in contour of the maxillary alveolar process under immediate dentures. A longitudinal clinical and x-ray cephalometric study covering 5 years. *Acta Odontol Scand*. 1967;25(1):45-75.

22. Tallgren A. The continuing reduction of the residual alveolar ridges in complete denture wearers: a mixed-longitudinal study covering 25 years. *J Prosthet Dent*. 2003;89(5):427-35.

23. Takahashi JM, Machado FM, Nuñez JM, Consani RL, Mesquita MF. Relining of prosthesis with auto-polymerizing hard denture reline resins: effect of post-polymerization treatment on flexural strength. *Gerodontology*. 2009;26(3):232–6
24. International Organization for Standardization. ISO20795-1(2013). Dentistry Part 1: Denture base polymers. Geneva, Switzerland 2013.
25. Powers JM, Sakaguchi RL. Craig's restorative dental materials. 12th ed. Missouri: Mosby; 2006 p. 539-40.
26. Ferracane J. Materials in dentistry: principles and applications. Pennsylvania: J.B. Lippincott; 1995 p. 262-3.
27. Odian G. Principles of polymerization. 4th ed. New York: John Wiley and Sons Ltd; 2004 p. 205-64.
28. Levin B. A reliable reline-rebase technique. *J Prosthet Dent*. 1976;36(2):219-25.
29. Hill EE, Rubel B. Direct chairside hard reline at delivery of a newly fabricated distal extension removable partial denture: considerations and techniques. *J Can Dent Assoc*. 2011;77:b84.
30. Barco MT, Moore BK, Swartz ML, Boone ME, Dykema RW, Phillips RW. The effect of relining on the accuracy and stability of maxillary complete dentures :An in vitro and in vivo study *J Prosthet Dent* 1979;42(1):17–22.
31. Polyzois GL, Lagouvardos PE, Frangou MJ. Flexural and bond strengths of relined denture polymers assessed by four-point bending tests and Weibull analysis. *Gerodontology*. 2012;29(2):543-52.
32. Zarb GA, Bolender CL. Prosthodontic treatment for edentulous patients: complete dentures and implant-supported prostheses. St. Louis: Mosby; 2003 p. 303-14.
33. Tucker KM. Relining complete dentures with the use of a functional impression. *J Prosthet Dent*. 1966;16(6):1054-57.
34. Vallittu PK, Ruyter IE, Buykuilmaz S. Effect of polymerization temperature and time on the residual monomer content of denture base polymers. *Eur J Oral Sci*. 1998;106(1):588-93.
35. Wyatt CC, Harrop TJ, MacEntee MI. A comparison of physical characteristics of six

hard denture relining materials. *J Prosthet Dent*. 1986;55(3):343-6.

36. Arima T, Murata H, Hamada T. Analysis of composition and structure of hard autopolymerizing relining resins. *J Oral Rehabil*. 1996;23(5):346-52.

37. Cucci AL, Giampaolo ET, Leonardi P, Vergani CE. Unrestricted linear dimensional changes of two hard chairside relining resins and one heat-curing acrylic resin. *J Prosthet Dent*. 1996;76(4):414-7.

38. Kedjarune U, Charoenworraluk N, Koontongkaew S. Release of methyl methacrylate from heat-cured and autopolymerized resins: cytotoxicity testing related to residual monomer. *Aust Dent J*. 1999;44(1):25-30.

39. Chaves CA, Machado AL, Vergani CE, de Souza RF, Giampaolo ET. Cytotoxicity of denture base and hard chairside relining materials: a systematic review. *J Prosthet Dent*. 2012;107(2):114-27.

40. Lai YL, Chen YT, Lee SY, Shieh TM, Hung SL. Cytotoxic effects of dental resin liquids on primary gingival fibroblasts and periodontal ligament cells in vitro. *J Oral Rehabil*. 2004;31(12):1165-72.

41. Mutluay MM, Ruyter IE. Evaluation of adhesion of chairside hard relining materials to denture base polymers. *J Prosthet Dent*. 2005;94(5):445-52.

42. Tanasamanchoke C, Wiwatwarrapan C. Increased wetting time of methyl formate-methyl acetate did not increase tensile bond strength of relined denture base resin. *CU Dent J*. 2015;38(Suppl):75-82.

43. Hout D, Wonglamsam A, Kanchanasavita W. Flexural strength of relined denture base using different thickness of self-cured relining material. *M Dent J*. 2017;37(2):223-32.

44. Stysiak ZD. Experimental investigations on the cytotoxic nature of methyl methacrylate. *J Prosthet Dent* 1980;44(1):13-6.

45. Matsumura H, Tanoue N, Kawasaki K, Atsuta M. Clinical evaluation of a chemically cured hard denture relining material. *J Oral Rehabil*. 2001;28(7):640-4.

46. Arima T, Murata H, Hamada T. Properties of highly cross-linked autopolymerizing relining acrylic resins. *J Prosthet Dent*. 1995;73(1):55-9.

47. McCabe JF, Walls AW. Applied dental materials. 8th ed: Wiley-Blackwell 1998 p.

108-14.

48. Yatabe M, Seki H, Shirasu N, Sone M. Effect of the reducing agent on the oxygen-inhibited layer of the cross-linked reline material. *J Oral Rehabil.* 2001;28(2):180-5.
49. Vallittu PK. The effect of surface treatment of denture acrylic resin on the residual monomer content and its release into water. *Acta Odontol Scand.* 1996;54(3):188-92.
50. Vergani CE, Seo RS, Pavarina AC, dos Santos Nunes Reis JM. Flexural strength of autopolymerizing denture reline resins with microwave postpolymerization treatment. *J Prosthet Dent.* 2005;93(6):577-83.
51. Patil P, Ramesh C, Mandokar R. Comparison of residual monomer content and flexural strength of two auto-polymerizing reline resins subjected to microwave post-polymerization treatment. *J Indian Prosthodont Soc.* 2009;9(3):136-41.
52. Charasseangpaisarn T, Wiwatwarrapan C. The effect of various frequencies of ultrasonic cleaner in reducing residual monomer in acrylic resin. *Ultrasonics.* 2015;63:163-7.
53. Charasseangpaisarn T, Wiwatwarrapan C, Leklerssiriwong N. Ultrasonic cleaning reduces the residual monomer in acrylic resins. *J Dent Sci.* 2016;11(4):443-8.
54. Thaitammayanon C, Sirichompun P, Wiwatwarrapan C. Ultrasonic treatment reduced residual monomer in methyl methacrylate-based orthodontic base-plate material. *Dent Oral Craniofac Res* [Internet]. 2018; 4:doi: 10.15761/DOCR.1000251.
55. Wiwatwarrapan C, Kobnithikulwong N. Ultrasonic treatment in ethanol-water reduce residual monomer in acrylic resin. The 3rd Meeting of the International Association for Dental Research–APR; June 23-25, 2016. Soul, Korea p. 1900.
56. Arriwiratchakun P, Wiwatwarrapan C. Effect of ultrasonic treatment on the leachable monomer content from a self cured acrylic hard denture reline material. *J Dent Assoc Thai.* 2019;69(2):175-84.
57. Ogawa T, Tanaka M, Koyano K. Effect of water temperature during polymerization on strength of autopolymerizing resin. *J Prosthet Dent.* 2000;84(2):222-4.
58. Ewoldsen N, Koka S. Wiropress SL: A multipurpose dental pressure vessel with transparent chamber. *J Prosthodont* 1998;7(2):126-8.

59. Machado CV, Fortes CB, Collares FM, Forte MMC, Samuel SMW. Effect of different curing condition on material properties of acrylic resin for orthodontic appliances. *Orthod Waves*. 2010;69(1):18-22.
60. Donovan TE, Hurst RG, Campagni WV. Physical properties of acrylic resin polymerized by four different techniques. *J Prosthet Dent*. 1985;54(4):522-4.
61. Schulz GV, Henrici G. Reaktionskinetik der polymerisationshemmung durch molekularen sauerstoff. (Versuche mit methylmethacrylat). *Makromolekularische Chemie*. 1956;18(1):437-54.
62. Lamb DJ, Ellis B, Priestley D. The effects of process variables on levels of residual monomer in auto polymerizing dental acrylic resin. *J Dent*. 1983;11(1):80-8.
63. Kay CS, Kanchanavasita W, Wonglamsam A. Effect of three repairing materials on the flexural strength of repaired heat-cured acrylic resin denture base material. *M Dent J*. 2017;37(1):37-45.
64. Ogawa T, Hasegawa A. Effect of curing environment on mechanical properties and polymerizing behaviour of methyl-methacrylate autopolymerizing resin. *J Oral Rehabil*. 2005;32(3):221-6.
65. Lee SY, Lai YL, Hsu TS. Influence of polymerization conditions on monomer elution and microhardness of autopolymerized polymethyl methacrylate resin. *Eur J Oral Sci*. 2002;110(2):179-83. จุฬาลงกรณ์มหาวิทยาลัย
66. Machado AL, Bochio BC, Wady AF, Jorge JH, Canevarolo SV, Vergani CE. Impact strength of denture base and relined acrylic resins: an in vitro study. *J Dent Biomech* [Internet]. 2012; 3:1758736012459535. doi: 10.1177/1758736012459535.
67. Kungkapilas K, Santawisuk W. Effect of chemical surface treatment on bond strength of a heat-cured acrylic resin denture base and a relined resin. *CM Dent J*. 2014;35(1):51-61.
68. Reis JM, Pavarina AC, Giampaolo ET, Vergani CE, Machado AL, Erxleben J. Exothermic behavior, degree of conversion, and viscoelastic properties of experimental and commercially available hard chairside relined resins. *J Appl Polym Sci*. 2011;122(3):1669-76.

69. Kim SH, Watts DC. Exotherm behavior of the polymer-based provisional crown and fixed partial denture materials. *Dent Mater.* 2004;20(4):383-7.



Appendix

Table 12 One-sample Kolmogorov-Smirnov test analysis of the data distribution

Tests of Normality

	condition	Kolmogorov-Smirnov ^a			Shapiro-Wilk		
		Statistic	df	Sig.	Statistic	df	Sig.
Max stress	u control	.262	10	.050	.848	10	.055
	u air 10	.144	10	.200*	.977	10	.948
	u air 15	.234	10	.130	.836	10	.040
	u air 20	.261	10	.052	.868	10	.096
	u n2 10	.141	10	.200*	.942	10	.578
	u n2 15	.155	10	.200*	.948	10	.643
	u n2 20	.249	10	.080	.885	10	.148
	rebase control +H	.215	10	.200*	.903	10	.238
	rebase air10 +H	.137	10	.200*	.948	10	.642
	rebase air15 +H	.152	10	.200*	.925	10	.402
	rebase air20 +H	.152	10	.200*	.942	10	.572
	rebase n2 10 +H	.148	10	.200*	.942	10	.580
	rebase n2 15 +H	.192	10	.200*	.890	10	.169
	rebase n2 20 +H	.157	10	.200*	.942	10	.570
	rebase control -H	.213	10	.200*	.860	10	.076
	rebase air10 -H	.158	10	.200*	.973	10	.918
	rebase air15 -H	.198	10	.200*	.942	10	.572
	rebase air20 -H	.227	10	.155	.939	10	.540
rebase n2 10 -H	.123	10	.200*	.955	10	.730	
rebase n2 15 -H	.224	10	.167	.864	10	.084	
rebase n2 20 -H	.225	10	.163	.887	10	.158	

*. This is a lower bound of the true significance.

a. Lilliefors Significance Correction

Table 13 The Levene statistically analysis of the flexural strength of UT

Test of Homogeneity of Variances

Max stress

Levene Statistic	df1	df2	Sig.
2.171	6	63	.058

Table 14 The Levene statistically analysis of the flexural strength of TR+H

Test of Homogeneity of Variances

Max stress

Levene Statistic	df1	df2	Sig.
1.107	6	63	.369

Table 15 The Levene statistically analysis of the flexural strength of TR-H

Test of Homogeneity of Variances

Max stress

Levene Statistic	df1	df2	Sig.
2.110	6	63	.064

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Table 16 Levene statistically analysis of the flexural strength of groups control

Test of Homogeneity of Variances

Max stress

Levene Statistic	df1	df2	Sig.
2.110	6	63	.064

Table 17 The Levene statistically analysis of the flexural strength of groups air10 min

Test of Homogeneity of Variances

Max stress

Levene Statistic	df1	df2	Sig.
6.208	2	27	.006

Table 18 The Levene statistically analysis of the flexural strength of groups air15 min

Test of Homogeneity of Variances

Max stress

Levene Statistic	df1	df2	Sig.
5.446	2	27	.010

Table 19 The Levene statistically analysis of the flexural strength of groups air20 min

Test of Homogeneity of Variances

Max stress

Levene Statistic	df1	df2	Sig.
12.384	2	27	.000

Table 20 The Levene statistically analysis of the flexural strength of groups N₂10 min

Test of Homogeneity of Variances

Max stress

Levene Statistic	df1	df2	Sig.
3.859	2	27	.034

Table 21 The Levene statistically analysis of the flexural strength of groups N_2 15 min

Test of Homogeneity of Variances

Max stress

Levene Statistic	df1	df2	Sig.
3.486	2	27	.045

Table 22 The Levene statistically analysis of the flexural strength of groups N_2 20 min

Test of Homogeneity of Variances

Max stress

Levene Statistic	df1	df2	Sig.
5.165	2	27	.013

Table 23 One way ANOVA analysis of the flexural strength of UT

Multiple Comparisons

Dependent Variable: maxstress

	(I) condition	(J) condition	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
						Lower Bound	Upper Bound
Tukey HSD	u control	u air 10	-7.73900*	1.98193	.004	-13.7752	-1.7028
		u air 15	-8.27800*	1.98193	.002	-14.3142	-2.2418
		u air 20	-8.28700*	1.98193	.002	-14.3232	-2.2508
		u n2 10	-14.00700*	1.98193	.000	-20.0432	-7.9708
		u n2 15	-14.11700*	1.98193	.000	-20.1532	-8.0808
		u n2 20	-14.35100*	1.98193	.000	-20.3872	-8.3148
	u air 10	u control	7.73900*	1.98193	.004	1.7028	13.7752
		u air 15	-.53900	1.98193	1.000	-6.5752	5.4972
		u air 20	-.54800	1.98193	1.000	-6.5842	5.4882
		u n2 10	-6.26800*	1.98193	.037	-12.3042	-.2318
		u n2 15	-6.37800*	1.98193	.032	-12.4142	-.3418
		u n2 20	-6.61200*	1.98193	.023	-12.6482	-.5758

	u air 15	u control	8.27800*	1.98193	.002	2.2418	14.3142
		u air 10	.53900	1.98193	1.000	-5.4972	6.5752
		u air 20	-.00900	1.98193	1.000	-6.0452	6.0272
		u n2 10	-5.72900	1.98193	.074	-11.7652	.3072
		u n2 15	-5.83900	1.98193	.064	-11.8752	.1972
		u n2 20	-6.07300*	1.98193	.048	-12.1092	-.0368
	u air 20	u control	8.28700*	1.98193	.002	2.2508	14.3232
		u air 10	.54800	1.98193	1.000	-5.4882	6.5842
		u air 15	.00900	1.98193	1.000	-6.0272	6.0452
		u n2 10	-5.72000	1.98193	.075	-11.7562	.3162
		u n2 15	-5.83000	1.98193	.065	-11.8662	.2062
		u n2 20	-6.06400*	1.98193	.048	-12.1002	-.0278
	u n2 10	u control	14.00700*	1.98193	.000	7.9708	20.0432
		u air 10	6.26800*	1.98193	.037	.2318	12.3042
		u air 15	5.72900	1.98193	.074	-.3072	11.7652
		u air 20	5.72000	1.98193	.075	-.3162	11.7562
		u n2 15	-.11000	1.98193	1.000	-6.1462	5.9262
		u n2 20	-.34400	1.98193	1.000	-6.3802	5.6922
	u n2 15	u control	14.11700*	1.98193	.000	8.0808	20.1532
		u air 10	6.37800*	1.98193	.032	.3418	12.4142
		u air 15	5.83900	1.98193	.064	-.1972	11.8752
		u air 20	5.83000	1.98193	.065	-.2062	11.8662
		u n2 10	.11000	1.98193	1.000	-5.9262	6.1462
		u n2 20	-.23400	1.98193	1.000	-6.2702	5.8022
	u n2 20	u control	14.35100*	1.98193	.000	8.3148	20.3872
		u air 10	6.61200*	1.98193	.023	.5758	12.6482
		u air 15	6.07300*	1.98193	.048	.0368	12.1092
		u air 20	6.06400*	1.98193	.048	.0278	12.1002
		u n2 10	.34400	1.98193	1.000	-5.6922	6.3802
		u n2 15	.23400	1.98193	1.000	-5.8022	6.2702
Games-Howell	u control	u air 10	-7.73900*	2.11514	.032	-14.9621	-.5159
		u air 15	-8.27800*	2.23345	.026	-15.7795	-.7765
		u air 20	-8.28700	2.60629	.064	-16.8994	.3254

	u n2 10	-14.00700*	2.17739	.000	-21.3702	-6.6438
	u n2 15	-14.11700*	2.15958	.000	-21.4386	-6.7954
	u n2 20	-14.35100*	2.29364	.000	-22.0118	-6.6902
u air 10	u control	7.73900*	2.11514	.032	.5159	14.9621
	u air 15	-.53900	1.61034	1.000	-5.8843	4.8063
	u air 20	-.54800	2.09706	1.000	-7.7023	6.6063
	u n2 10	-6.26800*	1.53164	.010	-11.3367	-1.1993
	u n2 15	-6.37800*	1.50621	.008	-11.3591	-1.3969
	u n2 20	-6.61200*	1.69282	.016	-12.2543	-.9697
u air 15	u control	8.27800*	2.23345	.026	.7765	15.7795
	u air 10	.53900	1.61034	1.000	-4.8063	5.8843
	u air 20	-.00900	2.21634	1.000	-7.4469	7.4289
	u n2 10	-5.72900*	1.69128	.043	-11.3225	-.1355
	u n2 15	-5.83900*	1.66829	.034	-11.3603	-.3177
	u n2 20	-6.07300	1.83852	.050	-12.1527	.0067
u air 20	u control	8.28700	2.60629	.064	-.3254	16.8994
	u air 10	.54800	2.09706	1.000	-6.6063	7.7023
	u air 15	.00900	2.21634	1.000	-7.4289	7.4469
	u n2 10	-5.72000	2.15984	.179	-13.0171	1.5771
	u n2 15	-5.83000	2.14188	.160	-13.0847	1.4247
	u n2 20	-6.06400	2.27698	.169	-13.6638	1.5358
u n2 10	u control	14.00700*	2.17739	.000	6.6438	21.3702
	u air 10	6.26800*	1.53164	.010	1.1993	11.3367
	u air 15	5.72900*	1.69128	.043	.1355	11.3225
	u air 20	5.72000	2.15984	.179	-1.5771	13.0171
	u n2 15	-.11000	1.59246	1.000	-5.3727	5.1527
	u n2 20	-.34400	1.77000	1.000	-6.2113	5.5233
u n2 15	u control	14.11700*	2.15958	.000	6.7954	21.4386
	u air 10	6.37800*	1.50621	.008	1.3969	11.3591
	u air 15	5.83900*	1.66829	.034	.3177	11.3603
	u air 20	5.83000	2.14188	.160	-1.4247	13.0847
	u n2 10	.11000	1.59246	1.000	-5.1527	5.3727
	u n2 20	-.23400	1.74804	1.000	-6.0356	5.5676

u n2 20	u control	14.35100*	2.29364	.000	6.6902	22.0118
	u air 10	6.61200*	1.69282	.016	.9697	12.2543
	u air 15	6.07300	1.83852	.050	-.0067	12.1527
	u air 20	6.06400	2.27698	.169	-1.5358	13.6638
	u n2 10	.34400	1.77000	1.000	-5.5233	6.2113
	u n2 15	.23400	1.74804	1.000	-5.5676	6.0356

*. The mean difference is significant at the 0.05 level.

Table 24 One way ANOVA analysis of the flexural strength of TR+H

Multiple Comparisons

Dependent Variable: max stress

	(I) condition	(J) condition	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
						Lower Bound	Upper Bound
Tukey HSD	rebase control +H	rebase air10 +H	-2.36900*	.70495	.021	-4.5160	-.2220
		rebase air15 +H	-2.82400*	.70495	.003	-4.9710	-.6770
		rebase air20 +H	-2.76700*	.70495	.004	-4.9140	-.6200
		rebase n2 10 +H	-4.71500*	.70495	.000	-6.8620	-2.5680
		rebase n2 15 +H	-4.91000*	.70495	.000	-7.0570	-2.7630
		rebase n2 20 +H	-5.47400*	.70495	.000	-7.6210	-3.3270
	rebase air10 +H	rebase control +H	2.36900*	.70495	.021	.2220	4.5160
		rebase air15 +H	-.45500	.70495	.995	-2.6020	1.6920
		rebase air20 +H	-.39800	.70495	.998	-2.5450	1.7490
		rebase n2 10 +H	-2.34600*	.70495	.023	-4.4930	-.1990
		rebase n2 15 +H	-2.54100*	.70495	.011	-4.6880	-.3940
		rebase n2 20 +H	-3.10500*	.70495	.001	-5.2520	-.9580
	rebase air15 +H	rebase control +H	2.82400*	.70495	.003	.6770	4.9710
		rebase air10 +H	.45500	.70495	.995	-1.6920	2.6020
		rebase air20 +H	.05700	.70495	1.000	-2.0900	2.2040
		rebase n2 10 +H	-1.89100	.70495	.120	-4.0380	.2560
		rebase n2 15 +H	-2.08600	.70495	.062	-4.2330	.0610
		rebase n2 20 +H	-2.65000*	.70495	.007	-4.7970	-.5030

	rebase air20 +H rebase control +H	2.76700 [*]	.70495	.004	.6200	4.9140
	rebase air10 +H	.39800	.70495	.998	-1.7490	2.5450
	rebase air15 +H	-.05700	.70495	1.000	-2.2040	2.0900
	rebase n2 10 +H	-1.94800	.70495	.100	-4.0950	.1990
	rebase n2 15 +H	-2.14300	.70495	.051	-4.2900	.0040
	rebase n2 20 +H	-2.70700 [*]	.70495	.005	-4.8540	-.5600
	rebase n2 10 +H rebase control +H	4.71500 [*]	.70495	.000	2.5680	6.8620
	rebase air10 +H	2.34600 [*]	.70495	.023	.1990	4.4930
	rebase air15 +H	1.89100	.70495	.120	-.2560	4.0380
	rebase air20 +H	1.94800	.70495	.100	-.1990	4.0950
	rebase n2 15 +H	-.19500	.70495	1.000	-2.3420	1.9520
	rebase n2 20 +H	-.75900	.70495	.933	-2.9060	1.3880
	rebase n2 15 +H rebase control +H	4.91000 [*]	.70495	.000	2.7630	7.0570
	rebase air10 +H	2.54100 [*]	.70495	.011	.3940	4.6880
	rebase air15 +H	2.08600	.70495	.062	-.0610	4.2330
	rebase air20 +H	2.14300	.70495	.051	-.0040	4.2900
	rebase n2 10 +H	.19500	.70495	1.000	-1.9520	2.3420
	rebase n2 20 +H	-.56400	.70495	.984	-2.7110	1.5830
	rebase n2 20 +H rebase control +H	5.47400 [*]	.70495	.000	3.3270	7.6210
	rebase air10 +H	3.10500 [*]	.70495	.001	.9580	5.2520
	rebase air15 +H	2.65000 [*]	.70495	.007	.5030	4.7970
	rebase air20 +H	2.70700 [*]	.70495	.005	.5600	4.8540
	rebase n2 10 +H	.75900	.70495	.933	-1.3880	2.9060
	rebase n2 15 +H	.56400	.70495	.984	-1.5830	2.7110
Games-Howell	rebase control +H rebase air10 +H	-2.36900 [*]	.65609	.027	-4.5384	-.1996
	rebase air15 +H	-2.82400 [*]	.75653	.022	-5.3344	-.3136
	rebase air20 +H	-2.76700 [*]	.74778	.023	-5.2464	-.2876
	rebase n2 10 +H	-4.71500 [*]	.62931	.000	-6.8013	-2.6287
	rebase n2 15 +H	-4.91000 [*]	.73478	.000	-7.3437	-2.4763
	rebase n2 20 +H	-5.47400 [*]	.63145	.000	-7.5668	-3.3812
	rebase air10 +H rebase control +H	2.36900 [*]	.65609	.027	.1996	4.5384
	rebase air15 +H	-.45500	.73476	.995	-2.9023	1.9923

	rebase air20 +H	-.39800	.72574	.998	-2.8126	2.0166
	rebase n2 10 +H	-2.34600*	.60296	.015	-4.3405	-.3515
	rebase n2 15 +H	-2.54100*	.71234	.031	-4.9075	-.1745
	rebase n2 20 +H	-3.10500*	.60520	.001	-5.1065	-1.1035
rebase air15 +H	rebase control +H	2.82400*	.75653	.022	.3136	5.3344
	rebase air10 +H	.45500	.73476	.995	-1.9923	2.9023
	rebase air20 +H	.05700	.81768	1.000	-2.6451	2.7591
	rebase n2 10 +H	-1.89100	.71095	.172	-4.2733	.4913
	rebase n2 15 +H	-2.08600	.80581	.187	-4.7495	.5775
	rebase n2 20 +H	-2.65000*	.71285	.025	-5.0373	-.2627
rebase air20 +H	rebase control +H	2.76700*	.74778	.023	.2876	5.2464
	rebase air10 +H	.39800	.72574	.998	-2.0166	2.8126
	rebase air15 +H	-.05700	.81768	1.000	-2.7591	2.6451
	rebase n2 10 +H	-1.94800	.70163	.141	-4.2958	.3998
	rebase n2 15 +H	-2.14300	.79759	.158	-4.7788	.4928
	rebase n2 20 +H	-2.70700*	.70355	.019	-5.0600	-.3540
rebase n2 10 +H	rebase control +H	4.71500*	.62931	.000	2.6287	6.8013
	rebase air10 +H	2.34600*	.60296	.015	.3515	4.3405
	rebase air15 +H	1.89100	.71095	.172	-.4913	4.2733
	rebase air20 +H	1.94800	.70163	.141	-.3998	4.2958
	rebase n2 15 +H	-.19500	.68776	1.000	-2.4919	2.1019
	rebase n2 20 +H	-.75900	.57606	.835	-2.6625	1.1445
rebase n2 15 +H	rebase control +H	4.91000*	.73478	.000	2.4763	7.3437
	rebase air10 +H	2.54100*	.71234	.031	.1745	4.9075
	rebase air15 +H	2.08600	.80581	.187	-.5775	4.7495
	rebase air20 +H	2.14300	.79759	.158	-.4928	4.7788
	rebase n2 10 +H	.19500	.68776	1.000	-2.1019	2.4919
	rebase n2 20 +H	-.56400	.68972	.980	-2.8663	1.7383
rebase n2 20 +H	rebase control +H	5.47400*	.63145	.000	3.3812	7.5668
	rebase air10 +H	3.10500*	.60520	.001	1.1035	5.1065
	rebase air15 +H	2.65000*	.71285	.025	.2627	5.0373
	rebase air20 +H	2.70700*	.70355	.019	.3540	5.0600
	rebase n2 10 +H	.75900	.57606	.835	-1.1445	2.6625

rebase n2 15 +H	.56400	.68972	.980	-1.7383	2.8663
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*. The mean difference is significant at the 0.05 level.

Table 25 One way ANOVA analysis of the flexural strength of TR-H

Multiple Comparisons

Dependent Variable: max stress

	(I) condition	(J) condition	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
						Lower Bound	Upper Bound
Tukey HSD	rebase control -H	rebase air10 -H	-3.08000*	.70488	.001	-5.2268	-.9332
		rebase air15 -H	-3.45400*	.70488	.000	-5.6008	-1.3072
		rebase air20 -H	-3.55500*	.70488	.000	-5.7018	-1.4082
		rebase n2 10 -H	-5.35400*	.70488	.000	-7.5008	-3.2072
		rebase n2 15 -H	-5.39100*	.70488	.000	-7.5378	-3.2442
		rebase n2 20 -H	-5.84700*	.70488	.000	-7.9938	-3.7002
	rebase air10 -H	rebase control -H	3.08000*	.70488	.001	.9332	5.2268
		rebase air15 -H	-.37400	.70488	.998	-2.5208	1.7728
		rebase air20 -H	-.47500	.70488	.994	-2.6218	1.6718
		rebase n2 10 -H	-2.27400*	.70488	.031	-4.4208	-.1272
		rebase n2 15 -H	-2.31100*	.70488	.027	-4.4578	-.1642
		rebase n2 20 -H	-2.76700*	.70488	.004	-4.9138	-.6202
	rebase air15 -H	rebase control -H	3.45400*	.70488	.000	1.3072	5.6008
		rebase air10 -H	.37400	.70488	.998	-1.7728	2.5208
		rebase air20 -H	-.10100	.70488	1.000	-2.2478	2.0458
		rebase n2 10 -H	-1.90000	.70488	.116	-4.0468	.2468
		rebase n2 15 -H	-1.93700	.70488	.103	-4.0838	.2098
		rebase n2 20 -H	-2.39300*	.70488	.019	-4.5398	-.2462
	rebase air20 -H	rebase control -H	3.55500*	.70488	.000	1.4082	5.7018

		rebase air10 -H	.47500	.70488	.994	-1.6718	2.6218
		rebase air15 -H	.10100	.70488	1.000	-2.0458	2.2478
		rebase n2 10 -H	-1.79900	.70488	.159	-3.9458	.3478
		rebase n2 15 -H	-1.83600	.70488	.142	-3.9828	.3108
		rebase n2 20 -H	-2.29200*	.70488	.029	-4.4388	-.1452
	rebase n2 10 -H	rebase control - H	5.35400*	.70488	.000	3.2072	7.5008
		rebase air10 -H	2.27400*	.70488	.031	.1272	4.4208
		rebase air15 -H	1.90000	.70488	.116	-.2468	4.0468
		rebase air20 -H	1.79900	.70488	.159	-.3478	3.9458
		rebase n2 15 -H	-.03700	.70488	1.000	-2.1838	2.1098
		rebase n2 20 -H	-.49300	.70488	.992	-2.6398	1.6538
	rebase n2 15 -H	rebase control - H	5.39100*	.70488	.000	3.2442	7.5378
		rebase air10 -H	2.31100*	.70488	.027	.1642	4.4578
		rebase air15 -H	1.93700	.70488	.103	-.2098	4.0838
		rebase air20 -H	1.83600	.70488	.142	-.3108	3.9828
		rebase n2 10 -H	.03700	.70488	1.000	-2.1098	2.1838
		rebase n2 20 -H	-.45600	.70488	.995	-2.6028	1.6908
	rebase n2 20 -H	rebase control - H	5.84700*	.70488	.000	3.7002	7.9938
		rebase air10 -H	2.76700*	.70488	.004	.6202	4.9138
		rebase air15 -H	2.39300*	.70488	.019	.2462	4.5398
		rebase air20 -H	2.29200*	.70488	.029	.1452	4.4388
		rebase n2 10 -H	.49300	.70488	.992	-1.6538	2.6398
		rebase n2 15 -H	.45600	.70488	.995	-1.6908	2.6028
Games-Howell	rebase control -H	rebase air10 -H	-3.08000*	.56742	.001	-5.0278	-1.1322
		rebase air15 -H	-3.45400*	.56487	.001	-5.3968	-1.5112
		rebase air20 -H	-3.55500*	.61139	.000	-5.6051	-1.5049
		rebase n2 10 -H	-5.35400*	.91687	.000	-8.4388	-2.2692
		rebase n2 15 -H	-5.39100*	.73329	.000	-7.8150	-2.9670
		rebase n2 20 -H	-5.84700*	.78382	.000	-8.4461	-3.2479

rebase air10 -H	rebase control -	3.08000 [*]	.56742	.001	1.1322	5.0278
	H					
	rebase air15 -H	-.37400	.36553	.942	-1.5819	.8339
	rebase air20 -H	-.47500	.43397	.921	-1.9215	.9715
	rebase n2 10 -H	-2.27400	.80943	.158	-5.1487	.6007
	rebase n2 15 -H	-2.31100 [*]	.59351	.023	-4.3585	-.2635
	rebase n2 20 -H	-2.76700 [*]	.65491	.014	-5.0498	-.4842
rebase air15 -H	rebase control -	3.45400 [*]	.56487	.001	1.5112	5.3968
	H					
	rebase air10 -H	.37400	.36553	.942	-.8339	1.5819
	rebase air20 -H	-.10100	.43063	1.000	-1.5383	1.3363
	rebase n2 10 -H	-1.90000	.80764	.301	-4.7725	.9725
	rebase n2 15 -H	-1.93700	.59107	.068	-3.9800	.1060
	rebase n2 20 -H	-2.39300 [*]	.65270	.037	-4.6722	-.1138
rebase air20 -H	rebase control -	3.55500 [*]	.61139	.000	1.5049	5.6051
	H					
	rebase air10 -H	.47500	.43397	.921	-.9715	1.9215
	rebase air15 -H	.10100	.43063	1.000	-1.3363	1.5383
	rebase n2 10 -H	-1.79900	.84084	.388	-4.7202	1.1222
	rebase n2 15 -H	-1.83600	.63567	.118	-3.9766	.3046
	rebase n2 20 -H	-2.29200	.69335	.059	-4.6499	.0659
rebase n2 10 -H	rebase control -	5.35400 [*]	.91687	.000	2.2692	8.4388
	H					
	rebase air10 -H	2.27400	.80943	.158	-.6007	5.1487
	rebase air15 -H	1.90000	.80764	.301	-.9725	4.7725
	rebase air20 -H	1.79900	.84084	.388	-1.1222	4.7202
	rebase n2 15 -H	-.03700	.93323	1.000	-3.1639	3.0899
	rebase n2 20 -H	-.49300	.97344	.998	-3.7307	2.7447
rebase n2 15 -H	rebase control -	5.39100 [*]	.73329	.000	2.9670	7.8150
	H					
	rebase air10 -H	2.31100 [*]	.59351	.023	.2635	4.3585
	rebase air15 -H	1.93700	.59107	.068	-.1060	3.9800
	rebase air20 -H	1.83600	.63567	.118	-.3046	3.9766

	rebase n2 10 -H	.03700	.93323	1.000	-3.0899	3.1639
	rebase n2 20 -H	-.45600	.80290	.997	-3.1134	2.2014
rebase n2 20 -H	rebase control -H	5.84700*	.78382	.000	3.2479	8.4461
	rebase air10 -H	2.76700*	.65491	.014	.4842	5.0498
	rebase air15 -H	2.39300*	.65270	.037	.1138	4.6722
	rebase air20 -H	2.29200	.69335	.059	-.0659	4.6499
	rebase n2 10 -H	.49300	.97344	.998	-2.7447	3.7307
	rebase n2 15 -H	.45600	.80290	.997	-2.2014	3.1134

*. The mean difference is significant at the 0.05 level.



Table 26 One way ANOVA analysis of the flexural strength of groups control

Multiple Comparisons

Dependent Variable: maxstress

	(I) condition	(J) condition	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
						Lower Bound	Upper Bound
Tukey HSD	u control	rebase control +H	18.90700*	1.61651	.000	14.8990	22.9150
		rebase control -H	20.30400*	1.61651	.000	16.2960	24.3120
	rebase control +H	u control	-18.90700*	1.61651	.000	-22.9150	-14.8990
		rebase control -H	1.39700	1.61651	.667	-2.6110	5.4050
	rebase control -H	u control	-20.30400*	1.61651	.000	-24.3120	-16.2960
		rebase control +H	-1.39700	1.61651	.667	-5.4050	2.6110
Games-Howell	u control	rebase control +H	18.90700*	1.91466	.000	13.6758	24.1382
		rebase control -H	20.30400*	1.92046	.000	15.0663	25.5417
	rebase control +H	u control	-18.90700*	1.91466	.000	-24.1382	-13.6758
		rebase control -H	1.39700	.69654	.140	-.3810	3.1750
	rebase control -H	u control	-20.30400*	1.92046	.000	-25.5417	-15.0663
		rebase control +H	-1.39700	.69654	.140	-3.1750	.3810

*. The mean difference is significant at the 0.05 level.

Table 27 One way ANOVA analysis of the flexural strength of groups air10 min

Multiple Comparisons

Dependent Variable: maxstress

	(I) condition	(J) condition	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
						Lower Bound	Upper Bound
Tukey HSD	u air 10	rebase air10 +H	24.27700*	.93330	.000	21.9630	26.5910
		rebase air10 -H	24.96300*	.93330	.000	22.6490	27.2770
	rebase air10 +H	u air 10	-24.27700*	.93330	.000	-26.5910	-21.9630
		rebase air10 -H	.68600	.93330	.745	-1.6280	3.0000
	rebase air10 -H	u air 10	-24.96300*	.93330	.000	-27.2770	-22.6490
		rebase air10 +H	-.68600	.93330	.745	-3.0000	1.6280
Games-Howell	u air 10	rebase air10 +H	24.27700*	1.11280	.000	21.3187	27.2353
		rebase air10 -H	24.96300*	1.05242	.000	22.0862	27.8398
	rebase air10 +H	u air 10	-24.27700*	1.11280	.000	-27.2353	-21.3187
		rebase air10 -H	.68600	.51696	.403	-.6615	2.0335
	rebase air10 -H	u air 10	-24.96300*	1.05242	.000	-27.8398	-22.0862
		rebase air10 +H	-.68600	.51696	.403	-2.0335	.6615

* . The mean difference is significant at the 0.05 level.

Table 28 One way ANOVA analysis of the flexural strength of groups air15 min

Multiple Comparisons

Dependent Variable: maxstress

	(I) condition	(J) condition	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
						Lower Bound	Upper Bound
Tukey HSD	u air 15	rebase air15 +H	24.36100 [*]	1.14313	.000	21.5267	27.1953
		rebase air15 -H	25.12800 [*]	1.14313	.000	22.2937	27.9623
	rebase air15 +H	u air 15	-24.36100 [*]	1.14313	.000	-27.1953	-21.5267
		rebase air15 -H	.76700	1.14313	.782	-2.0673	3.6013
	rebase air15 -H	u air 15	-25.12800 [*]	1.14313	.000	-27.9623	-22.2937
		rebase air15 +H	-.76700	1.14313	.782	-3.6013	2.0673
Games-Howell	u air 15	rebase air15 +H	24.36100 [*]	1.37650	.000	20.7183	28.0037
		rebase air15 -H	25.12800 [*]	1.27249	.000	21.6254	28.6306
	rebase air15 +H	u air 15	-24.36100 [*]	1.37650	.000	-28.0037	-20.7183
		rebase air15 -H	.76700	.63737	.473	-.9273	2.4613
	rebase air15 -H	u air 15	-25.12800 [*]	1.27249	.000	-28.6306	-21.6254
		rebase air15 +H	-.76700	.63737	.473	-2.4613	.9273

*. The mean difference is significant at the 0.05 level.

Table 29 One way ANOVA analysis of the flexural strength of groups air20 min

Multiple Comparisons

Dependent Variable: maxstress

	(I) condition	(J) condition	Mean Difference (I- J)	Std. Error	Sig.	95% Confidence Interval	
						Lower Bound	Upper Bound
Tukey HSD	u air 20	rebase air20 +H	24.42700*	1.59292	.000	20.4775	28.3765
		rebase air20 -H	25.03600*	1.59292	.000	21.0865	28.9855
	rebase air20 +H	u air 20	-24.42700*	1.59292	.000	-28.3765	-20.4775
		rebase air20 -H	.60900	1.59292	.923	-3.3405	4.5585
	rebase air20 -H	u air 20	-25.03600*	1.59292	.000	-28.9855	-21.0865
		rebase air20 +H	-.60900	1.59292	.923	-4.5585	3.3405
Games- Howell	u air 20	rebase air20 +H	24.42700*	1.91990	.000	19.2230	29.6310
		rebase air20 -H	25.03600*	1.86504	.000	19.8921	30.1799
	rebase air20 +H	u air 20	-24.42700*	1.91990	.000	-29.6310	-19.2230
		rebase air20 -H	.60900	.66917	.643	-1.1315	2.3495
	rebase air20 -H	u air 20	-25.03600*	1.86504	.000	-30.1799	-19.8921
		rebase air20 +H	-.60900	.66917	.643	-2.3495	1.1315

*. The mean difference is significant at the 0.05 level.

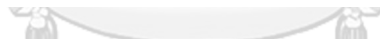


Table 30 One way ANOVA analysis of the flexural strength of groups N₂10 min

Multiple Comparisons

Dependent Variable: maxstress

	(I) condition	(J) condition	Mean Difference (I- J)	Std. Error	Sig.	95% Confidence Interval		
						Lower Bound	Upper Bound	
Tukey HSD	u n2 10	rebase n2 10 +H	28.19900*	1.17135	.000	25.2947	31.1033	
		rebase n2 10 -H	28.95700*	1.17135	.000	26.0527	31.8613	
	rebase n2 10 +H	u n2 10	-28.19900*	1.17135	.000	-31.1033	-25.2947	
		rebase n2 10 -H	.75800	1.17135	.796	-2.1463	3.6623	
	rebase n2 10 -H	u n2 10	-28.95700*	1.17135	.000	-31.8613	-26.0527	
		rebase n2 10 +H	-.75800	1.17135	.796	-3.6623	2.1463	
	Games- Howell	u n2 10	rebase n2 10 +H	28.19900*	1.21291	.000	24.9331	31.4649
			rebase n2 10 -H	28.95700*	1.37605	.000	25.4002	32.5138
rebase n2 10 +H		u n2 10	-28.19900*	1.21291	.000	-31.4649	-24.9331	
		rebase n2 10 -H	.75800	.86689	.665	-1.5170	3.0330	
rebase n2 10 -H		u n2 10	-28.95700*	1.37605	.000	-32.5138	-25.4002	
		rebase n2 10 +H	-.75800	.86689	.665	-3.0330	1.5170	

*. The mean difference is significant at the 0.05 level.



Table 31 One way ANOVA analysis of the flexural strength of groups N_215min

Multiple Comparisons

Dependent Variable: maxstress

	(I) condition	(J) condition	Mean	Std. Error	Sig.	95% Confidence Interval	
			Difference (I-J)			Lower Bound	Upper Bound
Tukey HSD	u n2 15	rebase n2 15 +H	28.11400*	1.10205	.000	25.3816	30.8464
		rebase n2 15 -H	29.03000*	1.10205	.000	26.2976	31.7624
	rebase n2 15 +H	u n2 15	-28.11400*	1.10205	.000	-30.8464	-25.3816
		rebase n2 15 -H	.91600	1.10205	.687	-1.8164	3.6484
	rebase n2 15 -H	u n2 15	-29.03000*	1.10205	.000	-31.7624	-26.2976
		rebase n2 15 +H	-.91600	1.10205	.687	-3.6484	1.8164
Games- Howell	u n2 15	rebase n2 15 +H	28.11400*	1.24007	.000	24.8473	31.3807
		rebase n2 15 -H	29.03000*	1.23018	.000	25.7802	32.2798
	rebase n2 15 +H	u n2 15	-28.11400*	1.24007	.000	-31.3807	-24.8473
		rebase n2 15 -H	.91600	.76970	.474	-1.0487	2.8807
	rebase n2 15 -H	u n2 15	-29.03000*	1.23018	.000	-32.2798	-25.7802
		rebase n2 15 +H	-.91600	.76970	.474	-2.8807	1.0487

*. The mean difference is significant at the 0.05 level.



Table 32 One way ANOVA analysis of the flexural strength of groups N₂20 min

Multiple Comparisons

Dependent Variable: maxstress

	(I) condition	(J) condition	Mean	Std. Error	Sig.	95% Confidence Interval		
			Difference (I-J)			Lower Bound	Upper Bound	
Tukey HSD	u n2 20	rebase n2 20 +H	27.78400*	1.25279	.000	24.6778	30.8902	
		rebase n2 20 -H	28.80800*	1.25279	.000	25.7018	31.9142	
	rebase n2 20	u n2 20	-27.78400*	1.25279	.000	-30.8902	-24.6778	
	+H	rebase n2 20 -H	1.02400	1.25279	.696	-2.0822	4.1302	
	rebase n2 20 -	u n2 20	-28.80800*	1.25279	.000	-31.9142	-25.7018	
	H	rebase n2 20 +H	-1.02400	1.25279	.696	-4.1302	2.0822	
	Games-Howell	u n2 20	rebase n2 20 +H	27.78400*	1.41194	.000	23.9511	31.6169
			rebase n2 20 -H	28.80800*	1.47884	.000	24.8806	32.7354
rebase n2 20		u n2 20	-27.78400*	1.41194	.000	-31.6169	-23.9511	
+H		rebase n2 20 -H	1.02400	.72659	.360	-.8523	2.9003	
rebase n2 20 -		u n2 20	-28.80800*	1.47884	.000	-32.7354	-24.8806	
H		rebase n2 20 +H	-1.02400	.72659	.360	-2.9003	.8523	

*. The mean difference is significant at the 0.05 level.

Table 33 3-way ANOVA analysis of the flexural strength

Tests of Between-Subjects Effects

Dependent Variable: max stress

Source	Type III Sum of Squares	df	Mean Square	F	Sig.	Partial Eta Squared
Corrected Model	29016.130 ^a	17	1706.831	230.880	.000	.960
Intercept	611547.204	1	611547.204	82723.045	.000	.998
product	28300.714	2	14150.357	1914.097	.000	.959
atmosphere	562.259	1	562.259	76.056	.000	.319
time	7.630	2	3.815	.516	.598	.006
product * atmosphere	143.473	2	71.737	9.704	.000	.107
product * time	.220	4	.055	.007	1.000	.000
atmosphere * time	1.403	2	.702	.095	.910	.001
product * atmosphere * time	.430	4	.107	.015	1.000	.000
Error	1197.618	162	7.393			
Total	641760.953	180				
Corrected Total	30213.749	179				

a. R Squared = .960 (Adjusted R Squared = .956)



Table 34 Descriptive Statistics of 3-way ANOVA analysis of the flexural strength

Descriptive Statistics

Dependent Variable: max stress

product	atmosphere	time	Mean	Std. Deviation	N
Unifast	Air	10min	72.6240	3.22387	10
		15min	73.1630	3.94190	10
		20min	73.1720	5.79509	10
		Total	72.9863	4.30559	30
	Nitrogen	10min	78.8920	3.61467	10
		15min	79.0020	3.50618	10
		20min	79.2360	4.27353	10
		Total	79.0433	3.68229	30
	Total	10min	75.7580	4.63154	20
		15min	76.0825	4.70698	20
		20min	76.2040	5.85112	20
		Total	76.0148	5.01038	60
rebase+ H	Air	10min	48.3470	1.41064	10
		15min	48.8020	1.84630	10
		20min	48.7450	1.81028	10
		Total	48.6313	1.65375	30
	Nitrogen	10min	50.6930	1.28284	10
		15min	50.8880	1.75626	10
		20min	51.4520	1.29334	10
		Total	51.0110	1.44715	30
	Total	10min	49.5200	1.78058	20
		15min	49.8450	2.05448	20
		20min	50.0985	2.06713	20
		Total	49.8212	1.95278	60
rebase- H	Air	10min	47.6610	.82615	10
		15min	48.0350	.80847	10
		20min	48.1360	1.09581	10

		Total	47.9440	.91133	30
	Nitrogen	10min	49.9350	2.42266	10
		15min	49.9720	1.68523	10
		20min	50.4280	1.89909	10
		Total	50.1117	1.96828	30
	Total	10min	48.7980	2.11289	20
		15min	49.0035	1.62550	20
		20min	49.2820	1.91301	20
		Total	49.0278	1.87271	60
Total	Air	10min	56.2107	11.97830	30
		15min	56.6667	12.12192	30
		20min	56.6843	12.34846	30
		Total	56.5206	12.01524	90
	Nitrogen	10min	59.8400	13.93678	30
		15min	59.9540	13.90925	30
		20min	60.3720	13.84007	30
		Total	60.0553	13.74034	90
	Total	10min	58.0253	13.01321	60
		15min	58.3103	13.04096	60
		20min	58.5282	13.13613	60
		Total	58.2879	12.99199	180

Table 35 Descriptive Statistics of One way ANOVA analysis of the flexural strength of products

Descriptive

Max stress

	N	Mean	Std. Deviation	Std. Error	95% Confidence Interval for Mean		Minimum	Maximum
					Lower Bound	Upper Bound		
Unifast	60	76.0148	5.01038	.64684	74.7205	77.3092	60.50	85.80
rebase+ H	60	49.8212	1.95278	.25210	49.3167	50.3256	46.12	53.51
rebase- H	60	49.0278	1.87271	.24177	48.5441	49.5116	45.81	53.57
Total	180	58.2879	12.99199	.96837	56.3771	60.1988	45.81	85.80

Table 36 One way ANOVA analysis of the flexural strength of products

Multiple Comparisons

Dependent Variable: max stress

	(I) product	(J) product	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
						Lower Bound	Upper Bound
Tukey HSD	Unifast	rebase+ H	26.19367 [*]	.60023	.000	24.7750	27.6124
		rebase- H	26.98700 [*]	.60023	.000	25.5683	28.4057
	rebase+ H	Unifast	-26.19367 [*]	.60023	.000	-27.6124	-24.7750
		rebase- H	.79333	.60023	.385	-.6254	2.2120
	rebase- H	Unifast	-26.98700 [*]	.60023	.000	-28.4057	-25.5683
		rebase+ H	-.79333	.60023	.385	-2.2120	.6254
Games-Howell	Unifast	rebase+ H	26.19367 [*]	.69423	.000	24.5344	27.8530
		rebase- H	26.98700 [*]	.69054	.000	25.3359	28.6381
	rebase+ H	Unifast	-26.19367 [*]	.69423	.000	-27.8530	-24.5344
		rebase- H	.79333	.34929	.064	-.0358	1.6225
	rebase- H	Unifast	-26.98700 [*]	.69054	.000	-28.6381	-25.3359
		rebase+ H	-.79333	.34929	.064	-1.6225	.0358

*. The mean difference is significant at the 0.05 level.

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