COLOR STABILITY, NANOHARDNESS AND ELASTIC MODULUS OF DENTURE BASE ACRYLIC RESINS WITH VARIOUS POLYMERIZATION TECHNIQUES

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A Thesis Submitted in Partial Fulfillment of the Requirements
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เสถียรภาพของสี ความแข็งผิวระดับนาโน และ ค่าโมดูลัสยืดหยุ่นของอะคริลิกเรซินสำหรับทำฐานฟันเทียมที่มีกระบวนการพอลิเมอไรเซชันที่แตกต่างกัน

น.ส.ศวิตา โล่ศุภกาญจน์

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ศวิตา โล่ศุภกาญจน์ : เสถียรภาพของสี ความแข็งผิวระดับนาโน และ ค่าโมดูลัสยืดหยุ่นของอะคริลิกเรซินสำหรับทำฐานฟันเทียมที่มีกระบวนการพอลิเมอไรเซชันที่แตกต่างกัน. (COLOR STABILITY, NANOHARDNESS AND ELASTIC MODULUS OF DENTURE BASE ACRYLIC RESINS WITH VARIOUS POLYMERIZATION TECHNIQUES) อ.ที่ปรึกษาหลัก : รศ. ทพ. ดร.แมนสรวง อักษรนุกิจ

วัตถุประสงค์ของงานวิจัย เพื่อศึกษาเสถียรภาพของสี ความแข็งผิวระดับนาโน และ ค่าโมดูลัสยืดหยุ่นของอะคริลิกเรซินสำหรับทำฐานฟันเทียมที่มีกระบวนการพอลิเมอไรเซชันที่แตกต่างกันหลังจากนำตัวอย่างแช่ในกาแฟ น้ำกลั่นหรือเก็บในที่มืด โดยเตรียมชิ้นตัวอย่างรูปเหรียญ (9 X 2 มม.) จำนวน 90 ชิ้นด้วยอะคริลิกเรซินชนิดบ่มตัวด้วยความร้อน (TRX) รังสีไมโครเวฟ (BTC) และแสง (TRD) และวัดค่าความเปลี่ยนแปลงสีด้วยเครื่องสเปคโตโฟโตมิเตอร์ที่เวลา 1 7 28 และ 56 วัน จากนั้นสุ่มชิ้นตัวอย่าง 3 ชิ้นต่อ 1 กลุ่ม เพื่อทดสอบความแข็งผิวระดับนาโนและค่าโมดูลัสยืดหยุ่น 3 ครั้งต่อชิ้นงานในวันที่ 1 และ 56 ทำการวิเคราะห์ทางสถิติด้วยความแปรปรวนแบบผสมซ้ำตามด้วยการวิเคราะห์การเปรียบเทียบหลากหลายชนิดทูกีเอชเอสดีและการทดสอบความแตกต่างค่ากลางของสองประชากรไม่อิสระที่ระดับความเชื่อมั่นร้อยละ 95 ผลการศึกษายืนยันความสัมพันธ์ระหว่างชนิดของวัสดุอะคลิริกเรซิน สิ่งแวดล้อมสำหรับเก็บชิ้นศึกษาและเวลาต่อค่าเสถียรภาพสี (p<0.001) อะคริลิกเรซินทุกกลุ่มในกาแฟมีการเปลี่ยนสีมากที่สุดตั้งแต่วันที่ 7 และพบว่า TRD มีค่ามากที่สุด ตามด้วย BTC และ TRX ทั้งกลุ่มที่แช่ในกาแฟและน้ำกลั่น สีของอะคริลิกเรซินในกาแฟมีแนวโน้มมืดลง สีแดงน้อยลง และสีเหลืองมากขึ้น นอกจากนี้ไม่พบความสัมพันธ์ระหว่างสามปัจจัยต่อความแข็งผิวระดับนาโนและค่าโมดูลัสยืดหยุ่นอย่างมีนัยสำคัญทางสถิติ (p=0.103, 0.138) กลุ่ม TRX มีค่าแข็งผิวระดับนาโนและค่าโมดูลัสยืดหยุ่นต่ำที่สุด ในขณะที่ BTC และ TRD มีค่าใกล้เคียงกัน กลุ่มตัวอย่างทุกกลุ่มในกาแฟและน้ำกลั่นมีความแข็งผิวระดับนาโนและค่าโมดูลัสยืดหยุ่นลดลงหลังจากแช่ 56 วัน และค่าดังกล่าวในกาแฟมีค่าลดลงมากกว่าในน้ำกลั่น

สาขาวิชา	ทันตกรรมประดิษฐ์	ลายมือชื่อนิสิต ....
ปีการศึกษา	2562	ลายมือชื่อ อ.ที่ปรึกษาหลัก ....

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KEYWORD: Acrylic resin, Denture base, color stability, Nanohardness, Elastic modulus

Sawita Losuphakarn : COLOR STABILITY, NANOHARDNESS AND ELASTIC MODULUS OF DENTURE BASE ACRYLIC RESINS WITH VARIOUS POLYMERIZATION TECHNIQUES. Advisor: Assoc. Prof. MANSUANG ARKSORNNUKIT, D.D.S., M.S., Ph.D.

The objective of this study was to evaluate color stability, nanohardness and elastic modulus of denture base acrylic resins with various polymerization techniques after storing in coffee, distilled water or dark chamber. Ninety disc-shaped (9X2 mm) specimens of heat-cured (TRX), microwave-cured (BTC) and light-cured (TRD) were fabricated, and randomly divided in three conditions (n=10). The color difference (DE*) was measured using a spectrophotometer at 1, 7, 28 and 56 days; The nanohardness and the elastic modulus were measured from three randomly selected of each group at day 1 and day 56 after color measurement. The repeated mixed analysis of variance following with Tukey's HSD multiple comparison and paired samples t-test were statistically analyzed at the confidence level of 95%. The interaction among material groups, storage media over the period of time were observed in the DE* ($p<0.001$). The DE* of the coffee storage group showed the greatest color change since day 7; TRD was greater color change than BTC and TRX both in coffee and distilled water. All specimens in coffee storage group were darker, less red and more yellow. Moreover, the interaction among three factors of nanohardness and elastic modulus showed no significant difference ($p=0.103$, 0.138). Those of TRX demonstrated the lowest nanohardness and elastic modulus while BTC and TRD showed almost similar. The nanohardness and the elastic modulus of coffee storage group showed the greatest reduction at 56-day storage, following by those in water.

Field of Study: Prosthodontics		Student's Signature ....
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Sawita Losuphakarn

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CHAPTER I

INTRODUCTION

Background and Rationale

The worldwide increase of the over 60-year-old population in 2025 was estimated by WHO organization in the different increasing population proportion of each country. (1, 2) Several public health reports predicted that the aging population in Thailand was also approximately 10% in 2010, and then will increase to be 30% in 2050 (3), and 20% in 2025. (4) The most common oral health problems in the elderly are tooth loss, dental caries, periodontitis, dry mouth and oral cancer. (1) The tooth loss is one of the crucial oral health problems in the elderly (4, 5) and their quality of life including physical, psychological and social performances. (4) The oral health survey in Thailand in 2017 illustrated the 8.1% of the stage of losing natural teeth, edentulism, in the over 60-year-old population. (5) Therefore, the removable partial prostheses are an alternative of tooth/teeth and adjacent tissue replacement for improving the chewing ability, phonetic and appearance. The removable prostheses consist of the artificial teeth which simulate the function of the lost natural teeth, and the denture base which replaces the alveolar ridge resorption, also increases the mechanical properties of the prostheses. (4, 6)

Regarding the denture base, the considerable materials are polymer based materials, for example acrylic resin, rubber, and metal alloys such as cobalt-chromium alloy. The acrylic resin has been chosen under the reasons of acceptable dimensional stability, physical properties, mechanical properties, the acceptable appearance to predict the duration of denture service and patient satisfaction and biocompatible. (7) The acrylic resin denture base materials have been classified by the various polymerization method following the ISO 20795-1: 2013 to be heat activator, chemical activator, light activator or microwave-length activator. (8) Each type of acrylic resin performs the different characteristics including physical and mechanical properties. One of the most important physical properties which predict the appearance are the color stability that the color changes in the prostheses might represented the lifetime of denture as an indication of damage and ageing of dentures. (7) Several studies reported the effect of color change when acrylic resins immersed in beverages such as coffee, tea, cola and red wine which were significantly changed.

(9-12) However, the color change was not significantly changed after immersing in the food colorants.(13) The porosities, excessive residual monomers, unfavorable surface roughness, also the physical property, such as the water sorption and water solubility, would have mentioned to be the causation of color change. (14)

The mechanical property of the acrylic resin denture base material is also the crucial to the survival of the prostheses. Several studies reported the flexural strength, flexural modulus, impact strength and yield distance of the acrylic resins which resisted to the masticating forces of normal occlusion. (15-17) The hardness which represents the resistance of the material to plastic deformation, and the elastic modulus which indicates the resistance of elastically deform when a force is applied, are considered. (18) Some studies reported the microhardness and elastic modulus of the several acrylic resins with various polymerization methods and compositions. (19-23) However, the nanohardness and elastic modulus which indirectly calculated from a nanoindentater are still rarely reported.

The color stability after a period of service and the mechanical properties, especially the nanohardness and elastic modulus might be the crucial keys to predict the longevity of the removable prostheses. Moreover, the effect of the various polymerization methods of the denture base acrylic resin is still not elucidated. Therefore, the objective of this research is to investigate the color stability, nanohardness and elastic modulus of denture base acrylic resins with various polymerization techniques.

Research Objective

To investigate the color stability, the nanohardness and the elastic modulus of denture base acrylic resins with various polymerization techniques when immersed in coffee or distilled water and kept in dark chamber.

Research Hypothesis

1. H_0: There would be no significant difference among the color change of various acrylic denture base materials when immersed in coffee and water, and kept in dark chamber.

H_a: There would be significant difference among the color change of various acrylic denture base materials when immersed in coffee and water, and kept in dark chamber.

2. H_0: There would be no significant difference among the nanohardness values of various acrylic denture base materials when immersed in coffee and water, and kept in dark chamber.

   H_a: There would be significant difference among the nanohardness values of various acrylic denture base materials when immersed in coffee and water, and kept in dark chamber.

3. H_0: There would be no significant difference among the elastic modulus of various acrylic denture base materials when immersed in coffee and water, and kept in dark chamber.

   H_a: There would be significant difference among the elastic modulus of various acrylic denture base materials when immersed in coffee and water, and kept in dark chamber.

Keywords

Acrylic resin, Denture base, Color stability, Nanohardness, Elastic modulus

Research design

Experimental study

Proposed benefits

The denture base acrylic resin materials have been considered to be the material of choice for replacing the destructive oral structure after tooth/teeth lost because their well appearance and acceptable mechanical properties. The materials have been developed their compositions and polymerization methods for the purpose of fabrication time while remained their mechanical properties and esthetics. Therefore, the color stability and mechanical properties of the acrylic resins, especially the nanohardness and the elastic modulus, might be under the influence of the various polymerization methods. The proposed benefits of this *in vitro* study was to select the acrylic resin materials with the different polymerization mode that are suitable to the prostheses workflow in the aspect of the esthetics and the mechanical properties, i.e. the nanohardness and the elastic modulus.

CHAPTER II

REVIEW LITERATURE

Basic chemistry and polymerization process of acrylic resin

Basically, the chemistry of methacrylate based materials which is one of the polymeric materials used in dental service is consist of the repeat the smallest units called “monomer/mer” to be a large linear molecule called “polymer”. The repeated same mers in the polymer is called “homopolymer”; whereas, the repeated different mers in the same polymeric structure is called “heteromer”. Other forms of polymer might be the combination between the main polymer molecules and different monomer called “copolymers”, and the same small sets of oligomers to bind another polymer molecule called “cross-linking polymer”. (18)

The acrylic resin is the general dental term referring to polymethyl methacrylate (PMMA, polymer) which is consisted of the repeated methyl methacrylate molecules (MMA, mer) as the main composition with/ without the additive copolymer and/or the additive fillers. The manufactures actually design the preparation of the acrylic resin to be powder and liquid. The powder contains the PMMA with highly reactive double bonds, the initiator such as benzoyl peroxide and the color pigments. The main composition of the liquid is the MMA with/without the cross-linking agent and an activator for initiation of the redox reaction, also the inhibitor for increasing the storage time. (18) (Figure 1a). Another preparation form of the acrylic resin is a light-cured acrylic resin which contains urethane dimethacrylate (UDMA), the polymeric cross-linking network molecules incorporating with a photo-initiator. (24) (Figure 1b).

Regarding the polymerization reaction of the PMMA, two basic reaction systems are available. The first system, called a redox reaction initiated system, combines the MMA residuals together to be the longer polymeric chains. The activation system might be heat (heat-cured acrylic resin) or free radical producing chemical substances as tertiary amine (chemical-cured acrylic resin). The second system is based on the photo-initiation reactions that the specific spectrum light in the combination with the free radical producing chemical substances as champhoroquinone-amine produces the free radical. (24) The polymerization of PMMA basically consist of the initiation, the propagation and the inhibition or the termination. The initiation is begun when the chemical groups with unshared electrons in the free radical molecules were produced from the reaction of a peroxide initiator and amine accelerator. The free radicals attack the double bonds of monomer molecules, and form the activated monomer molecules instead. The propagation occurs when the free radicals continuously attack the double bonds of other monomer molecules, and grow up the length of polymer chain. The termination depends on the presence of the inhibitors such as hydroquinone. The inhibitor is not only combine between the main polymeric linear structures and the initiators forming the branches and cross-links, but also diminishes the initiation rate to prolong shelf life. (25, 26) (Figure 2)

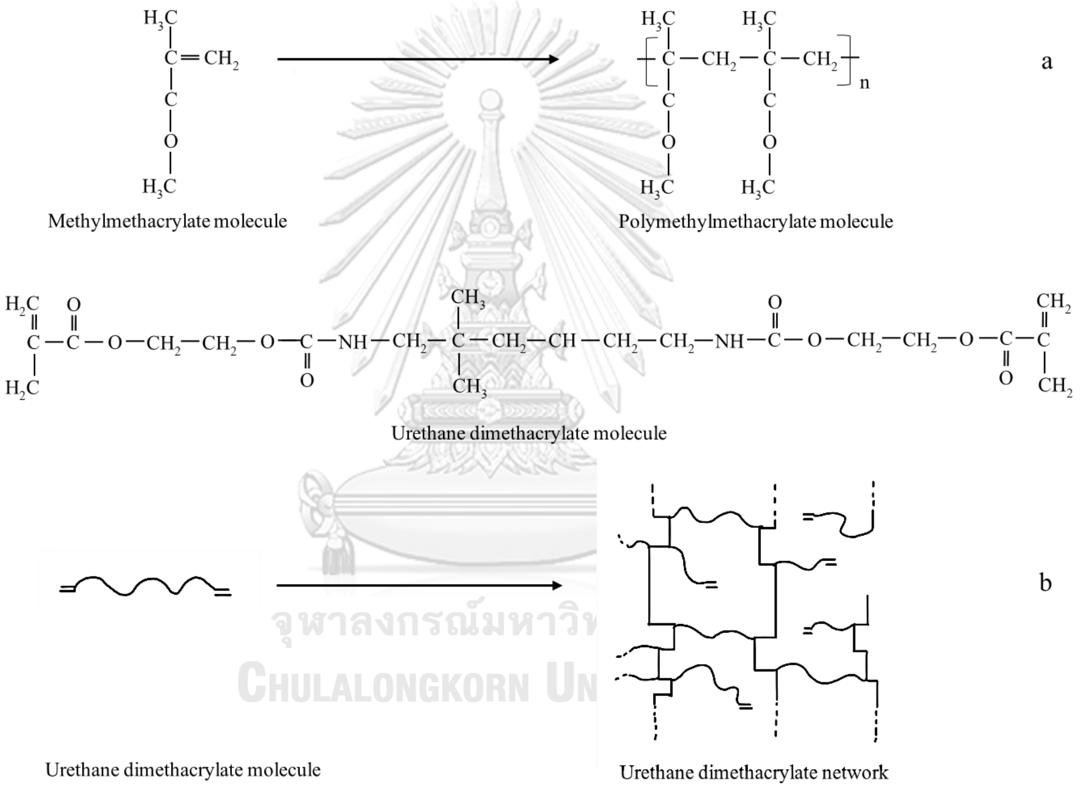


Figure 1 (a) The structure of methyl methacrylate (MMA) molecules as a monomer of Polymethylmethacrylate (PMMA), (b) The structure of Urethane dimethacrylate resin and the construction of dimethacrylate network

Modified from Cook WD, Beech DR, Tyas MJ. Structure and properties of methaerylate based dental restorative materials. Biomaterials 1985;6:362-68. and Anusavice KJ, Phillips RW, Shen C, Rawls HR. Phillips' Science of dental materials. 12^{th} ed. St. Louis: Elsevier Saunders; 2013.

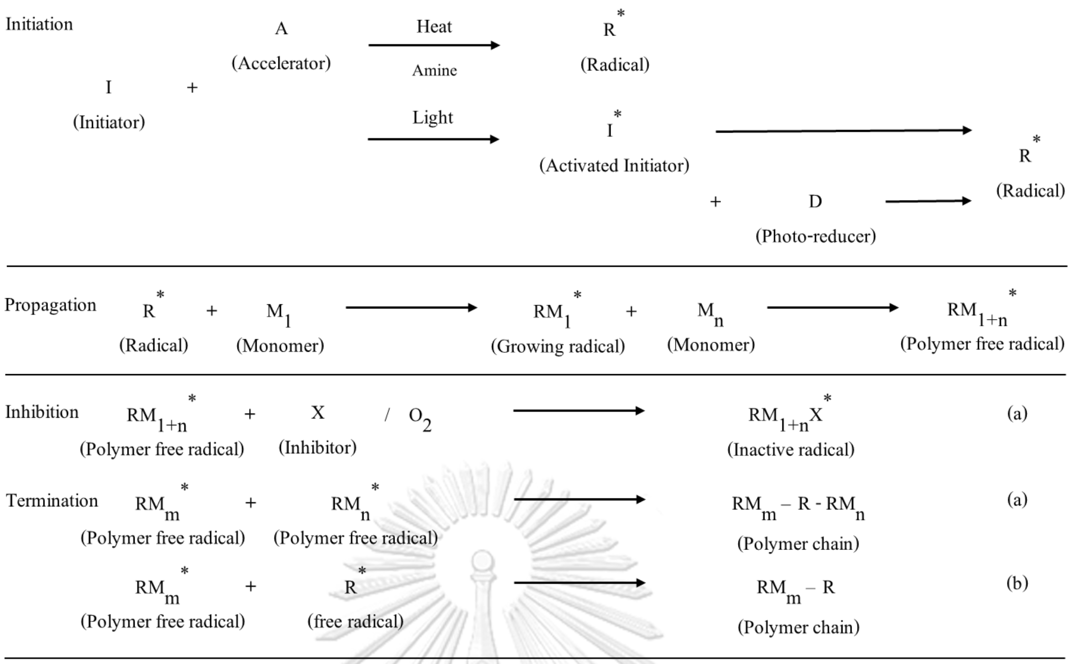


Figure 2 The polymerization reaction of the acrylic resin using heat, chemical and light activators

Modified from Wayne DC, Derrick RB, Beech JT, Martin JT. Structure and properties of methacrylate based dental restorative materials. Biomaterials 1985;6:362-68. (a) and O'Brieb WJ. Dental materials and their selection. 4th ed. Chicago: Quintessence; 2008. (b)

Denture base acrylic resins

According the development of the denture from the natural materials, for example wood, bone and ivory, were considered in the first era, but lack of esthetics, poor mechanical properties and ill-fitting fabrication which often irritated the oral tissues. Thereafter, many durable materials, for example the porcelain and gold, were used; however, the price and the unpredictable fabrication process were still the problems. The rubber materials such as, gutta percha, dry-heat vulcanization of rubber and vulcanite rubber were used as a standard denture material in 1936, and continuously used for over 75 years because these materials could produce the dentures exactly fitting the edentulous ridge with reasonable service price. The celluloid tortoise shell was also a material of choice which showed the good appearance rather than vulcanite rubber, but the bad taste, odor and dimensional instability during function were the disadvantages. Aluminum was also introduced instead of gold, but the bonding strength to the denture tooth was still re-evaluated. In the early of the 20th decade, the usage of Bakelite which is a phenol-formaldehyde resin with high dimensional stability and variation of colors but very technique sensitive. The metal alloys such as stainless steel, base metal alloys, also the vinyl resins as denture base materials was high cost and difficult fabrication. The PMMA was firstly introduced since 1937 by the invention of Dr. Walter Wright. That the better physical properties, acceptable mechanical properties and aesthetics, user friendly and biocompatibility have leaded the PMMA to be the material of choice. (27, 28)

The ISO 20795 (8) classified the acrylic resin denture base materials due to the various activation methods. (Table 1)

Heat-polymerized polymer is the worldwide used material for denture base material according to their acceptable physical and mechanical properties as well as their appearance. The optimal heat is required to initiate the polymerization reaction. The manufacturers produce their product to be 2 compositions, the powder with mainly PMMA, and the liquid consisting of mainly MMA, initiator (Benzoyl peroxide) and inhibitor (Hydroquinone). The Additive styrene butadiene rubber is included to improve impact strength of the material, called high impact strength resin. (Table 1) Compression molding technique and injection molding technique are available to fabricate the denture according to the product preparation.

Compression molding technique is a conventional prefabrication method which needs to make a plaster mold incorporation with a wax trial denture model, subsequently losses and washes the wax out. The mixture of the resin following the manufacturer recommendation, powder to liquid ratio is 23.4 g/ 10 mL, would be prepared until the dough stage, and then packed into the fabricated plaster mold after the separating media would be lightly painted over the plaster mold surface. (18) The hydraulic pressure equipment is needed to limit the volumetric polymerization shrinkage between 5-7%. (7, 18) On the other hand, the injection molding technique needs a special designed flask with two entrance holes that bind the wax pattern with wax sprues. After all wax pattern would be burnt out, the heated resin material would be injected into the mold under a special injecting machine. The study of polymerization shrinkage between two different technique reported that the comparative accuracy of denture base from two different fabrication technique was still questionable (18), but the other reported the greater dimensional stability and expansion of the injection molding technique comparing to the compression molding technique. (29)

The polymerization temperature is a crucial factor affecting on the mechanical properties of the denture base. The polymerization reaction of the acrylic resin material creating the temperature, called exothermic reaction. The suggested temperature would be above 70°C to form the free radicals from benzoyl peroxide but not over 100.8°C of the boiling point of monomer; otherwise, the greater amount of the residual monomer and porosities inside the denture bases would be observed. The guideline in table 2 suggests two curing process which the long cycle keeps constant temperature of 74°C for 8 hours, and which the short cycle keeps the temperature of 74°C for 2 hours, and then 100°C for 1 hour. For reducing the distortion of the denture base, the flasks should be slowly cooled down to the room temperature approximately 30 minutes, immersed in cool tap water before deflasked. (30)

Regarding the physical and mechanical properties of the heat-polymerized polymers, the less amount of residual monomer, low water sorption and solubility were reported comparing the auto-polymerized polymers under the condition of oral environment. (18) Poor fatigue resistance and flexural strength of the heat-polymerized resins were reported as seen in the clinical evidences of denture fracture especially at the midline of maxillary denture base. (31)

High impact strength resins were invented to increase the impact strength of acrylic resin denture base by adding the inclusion of the styrene/butadiene rubber. The other additive particles such as hydroxyapatite, alumina, zirconia and titanium particles were also considered to increase the mechanical strength. The greater flexural strength, impact strength, thermal conductivity and the less of polymerization shrinkage, water sorption have been reported. Nevertheless, the newest materials with the hybrid fibers, hybrid fillers or the combination of hybrid fiber and fillers might be an alternative denture base material that enhance the mechanical properties of acrylic resin. (32-34) One study reported that the additive fillers, for examples hydroxyapatite fillers, silica-based fillers, carbon family fillers, and the additive fibers, for example silanated glass fibers, polyamide fibers, polyethylene and polypropylene fibers, natural fibers etc., significantly improved mechanical properties of denture base. (34)

Table 1 the classification, compositions and curing process of denture base polymers.

Type	Class	Mode of polymerization	Preparation	Compositions	Curing process
1	**1**	Heat-polymerized polymers	Liquid and powder	Powder: PMMA, High impact strength resins, Additive styrene/butadiene rubber Liquid: MMA Initiator: Benzoyl peroxide Inhibitor: Hydroquinone	Long cycle: 74ºC/8 hr Short cycle: 74ºC/2 hr and then 100ºC/1 hr
	2	Heat-polymerized polymers	Plastic cake		
2	**1**	Auto-polymerized polymers	Liquid and powder	Powder: PMMA Liquid: MMA Initiator: Benzoyl peroxide Inhibitor: Hydroquinone	Generate polymerization by activator (tertiary amine) at room temperature
	2	Auto-polymerized polymers	Liquid and powder for pour-type resins		
3	**-**	Thermoplastic polymerized polymers	-	PMMA with nylon, polyamide, polycarbonate resin	Heat-press injection technique
4	**-**	Light-polymerized polymers	-	Urethane dimethacrylate PMMA - derivative material High molecular weight acrylic resin monomers Activator: Camphoroquinone-amine	Emission of visible light in the shorter blue 400 to 500 nm
5	**-**	Microwave-polymerized polymers	-	Powder: PMMA/PMEA, copolymer Liquid: MMA	Curing cycle of 3-5 minutes at 500-600 W by microwaves

Abbreviation. PMMA: Polymethylmethacrylate, MMA: Methylmathacrylate, PMEA: Polymethylethacrylate

Modified from the international organization for standardization. ISO 20795 Dentistry – Base polymers – Part 1: Denture base polymers. 2013. and Anusavice KJ, Phillips RW, Shen C, Rawls HR. Phillips' Science of dental materials. 12th ed. St. Louis: Elsevier Saunders; 2013.

Injection-molded thermoplastic resins have been considered to be used as an alternative choice of denture base polymeric resin which required a sophisticate machine to turn the large hard polymeric grains to be liquid form, and then pressed into a plater mold. The materials consist of the different molecular weight linear polymeric molecules which can commercially be classified to be 4 groups; the thermoplastic acetal polymer, the thermoplastic polycarbonate, the thermoacrylic and the nylon modified thermoplastic resin. The thermoplastic acetal polymers containing the copolymers demonstrate better long term stability, occlusal wear resistance and patient vertical dimension maintenance during the provisional prosthodontic treatment; whereas, the thermoplastic polycarbonates containing the bis-phenol-A carbonate polymeric chains are considered to fabricate a provisional crown or bridge excluding the partial denture framework. The thermoacrylic resin is suggested to produce the temporary crowns and thermal polymerized denture base. The nylon modified thermoplastic resin, e.g. nylon polyamides, which is introduced in 1950s, is formed by the condensation reactions between dibasic acid and diamine. Some modification of the latest was done by adding the reinforced the glass fiber for decreasing the water sorption and melting point. (7, 14, 35)

Several researchers reported the advantages of the thermoplastic resins over the conventional denture base materials. The thermoplastic resins showed the greater flexibility characterized by the lower relative ratio of the proportional limit to the ultimate flexural strength and the lower flexural moduli; therefore, the patients would feel easy to familiar to the dentures with the thermoplastic denture bases. (36, 37) The polyamide and the polycarbonate resin denture bases showed the greater impact strength. (37) However, the thermoplastic resins trended to undergo permanent deformation during mastication due to the lower flexural strength at the proportional limit and the lower elastic modulus. (17, 37) The greater porosities and the more susceptible to discoloration by daily food and drink were also reported. (38, 39)

Self-cured/Auto-polymerized polymer consists of the same chemical polymeric structure molecules as the heat-polymerized polymer. (Table 1) The additive tertiary amine as chemical activators, e.g. dimethyl-para-toluidine, initiate the polymerization reaction when the powder and liquid were mixed together, and gradually increased the viscosity from the sandy stage, the fibrous stage, to the dough stage. The products would be fabricated in this stage, and leaved them to rubber stage in the room temperature, and then set. Generally, the dentists or the dental technicians would quickly process at room temperature for the optimal working time. (7, 14) However, some studies reported the greater amount of leachable residual monomer rather than the heat-polymerized polymer due to the incomplete polymerization. The remaining incomplete polymerized residual monomer would irritate the surrounding tissue, and then compromise the biocompatibility of denture base resins. (40, 41) The mechanical properties of denture base resins, especially the transverse strength, the fatigue strength, the fracture toughness and the flexural modulus, would be affected from the presence of the unreacted monomer. (14, 41, 42) One study reported the approximately 80 % of the transverse strength, poor color stability and poor bond strength in the chemical-polymerized polymer comparing to the heat-polymerized polymer. (43)

Visible light-polymerized polymers have been classified to be an alternative denture base material for the patient with hypersensitive to the MMA-based materials. The main composition is urethane dimethacrylate matrix (UDMA) with hydrophilic functional group similar to resin composite restorative material. The polymerization reaction occurs when the photosensitizing agent, camphorquinone, was stimulated by the emission of high intensity visible light. The acrylic resin beads are added for strengthening the polymer. (7, 18)

Several studies reported the advantages of the light-cured resins. The fabrication process was familiar for the dentists or the dental technicians to handle with acceptable working time. The manufacturers claimed their products that were reduce the processing time, especially the loss wax technique, good biocompatibility, less bacteria adherence, good dimensional stability and high fitting accuracy. (44, 45) Moreover, the greater microhardness, transverse strength, flexural strength, tensile strength and elastic modulus were reported comparing to the conventional acrylic resin denture base materials. (15, 44, 46, 47) However, one study reported the disadvantages of the light-cured resins including the porosities which leaded to the formation of microcracks under loading and the poor bond strength between the denture teeth and the light-cured resins (48), even the pre-treatment conditioning liquid to promote the bonding was applied. (49)

Microwave-polymerized resins are one of the PMMA based resins which were polymerized under the non-metallic glass fiber- reinforced flask and a microwave producing machine. The asymmetrically polarized molecules of MMA are rapidly flipped over in the 500-600 Watts/cycle electromagnetic field for 3 minutes. The heat was generated due to the molecular friction to complete the polymerization reaction. (7, 18, 50) Comparing the conventional heat-cured resins, many studies reported the equal or better dimensional stability (51), the equal microhardness, transverse strength and flexural modulus (16, 42, 52-54), the greater residual monomer and the greater porosity during microwave processing of the dentures due to the short polymerizing cycle (50), higher shear bond strength than acrylic resin teeth (55) and poor color stability. (56)

Color stability of acrylic resins

The nature of color is referred to the visual sensation when the visible 400 to 800 nm-wave length electromagnetic radiation affected to an object, and then reflected to the eye. During the light projected to the object, some wavelength wave would be absorbed into the object, and the others would be reflected to the eye in term of the object color. The scientific color measurement systems used in dentistry are Munsell color order system and the commission International de I'Eclairage (CIE) Lab color system. (57-59)

1. Munsell color order system is the common used color measuring system in dentistry as the dimension of color. The visual measurement method classified the color depending on the factor of visual response to the light and observer experience. The system gives explanation of the color to be 3 parameters:

Hue refers to the name of color which could be distinguished one color family from another by the separately-wavelengths of radiant energy quality of the observer sensation as red, green, blue, etc., but white, gray and black are considered with no hue.

Value refers to the lightness of color determined by gray scale which indicated the darkness to brightness, low values refer to dark colors and high values to light colors.

Chroma refers to the strength of color as the intensity of hue which the change in value by adding gray color always theoretically decreases chroma. (57, 58)

2. CIE Lab color system initiated by the Commission International de I'Eclairage in 1931, which defined the standardization of three dimensional XYZ tristimulus values as the three primary colors of red, green, blue and all mixtures of these three colors with the standard light source and calculated the quantitative color values.

Moreover, the most common collecting and analyzing methods of the color stability tests are the colorimetric and the spectrophotometric measurement methods which allow a digital, quantitative and reproducible means of color evaluation. These measuring methods could eliminate the human interpretation of color difference by imitating the visual perception of the human eyes which consist of the illumination source, the object and the observer sensor. The colorimetric measurement can interpret the color change in the specific range of the human eyes perception under the entire wavelength of day light (380 – 780 nm); whereas, the spectrophotometric measurement can interpret the wider wavelength of color changes apart from the visual human perception. (59)

In the CIE Lab color system, the color difference was calculated as follows eq 1.

$$\Delta E^*_{ab}(L^*a^*b^*) = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2} \qquad \text{(eq 1)}$$

where ΔE^* refers to color difference between two colors under specified condition, ΔL^* refers to the difference of value (blackness to whiteness), Δa^* refers to the difference level of color in the range of red (+a*) and green (-a*) and Δb^* refers to the difference level of color in the range of yellow (+b*) and blue (-b*).

According to the National Bureau of Standards (NBS), the noticeable color differences (ΔE^*) were quantified with NBS units (NBS unit = ΔE^*x 0.92) from 1.5 to 3.0. The unacceptable ΔE^* is over 3.0. (60) Some studies reported that the 50% of the observer could recognize the ΔE^* greater than 1 (61), the ΔE^* greater than 2 could be perceptible, and ΔE^* greater than 3.7 could clinically unacceptable. (62)

The factors related to the color stability

Color stability is one of the common used parameters to evaluate the esthetics and to indicate the aging and the damage of the denture acrylic resin material after a period of service. Several factors effect on the color stability including the polymerization methods (43, 56, 63-68), the staining solutions (9-13, 64, 65, 70-75), the immersion period (9-12, 64, 65, 70-72), the denture base resins composition (9, 13, 34, 39, 76, 77) and the usage of denture cleansers. (9, 63, 64, 78)

1. Polymerization methods – Several studies have reported that the different polymerization activators, from heat, chemical, light or microwave, in denture base acrylic resins showed the different color stability. The past reports from many research centers still reported the controversy of the color stability under the different polymerization methods. The fast heat-polymerized acrylic resin showed the greatest color change rather than the chemical-polymerized acrylic resin and the light-polymerized acrylic resin respectively. (56, 63) Other studies reported the greater color change of the light-polymerized acrylic resin than the heat-polymerized acrylic resin under immersion of the staining solutions (64), and the conventional heat-cured acrylic resins showed better color stability than the self-cured acrylic resins. (43) The color change of the microwave-polymerized acrylic resin was greater than that of normal and fast setting heat-polymerized acrylic resins (56); however, another study reported the similar color change of the microwave-cured and that of the heat-cured acrylic resins, but lower than self-cured resins. (65) Each polymerization method and its environment condition were affected on the amount of leached out monomer from the deeper layer or the surface of the denture base resins (66, 67) and the greater degree of conversion in resin composite were reported the lower color change in water storage. (68)

2. Types of staining solutions – Many studies reported the effect of the staining solution on color change of denture base acrylic resins. (9-13, 64, 65, 69-72) Regarding the staining solution, the daily beverages such as coffee, tea, cola or red wine and the artificial stains are common considered. The coffee is the most chromogenic staining solution and the most famous beverage with mild acidity, used in the color stability test and the daily consumption of coffee in patient behavior were reported approximately to be 3.2 cups a day with 120 ml cup. (73, 74) Many studies used the coffee as the staining solution, and exhibited the significant effect of the color change of the acrylic resins rather than tea and distilled water over the observation periods. (10, 69-72) The others reported the greater color change of acrylic resin immersed in tea, organic juice and grape juice comparing to that immersed in coffee and cola. (12, 64, 69) Moreover, one studies used the food colorants reported the greater of color change in 3% erythrosine colorant group rather than the tartrazine and sunset yellow. (13)

3. Immersion periods – The influence of the immersion period on the color stability of the denture base acrylic resins under the staining solution have been reported. Several reports exhibited the increase color change after the longer immersion periods. (9-12, 64, 65, 69-72)

4. The composition of each types of denture base resins – The relationship of the acrylic resin compositions and the color stability have been reported. The conventional acrylic resin and the cross-linked acrylic resin showed the different color change after the immersion in the food colorants (13), the higher molecular weight and complicate molecular structure as the UDMA-based resin showed greater color stability than the PMMA-based resin. (75) The additive fillers into the denture base resins also affected on the color stability. The fiber-reinforced heat-polymerized acrylic resins exhibited more susceptible to discoloration comparing to the conventional heat-polymerized denture base material, but the rubber-reinforced high impact heat-polymerized denture base material showed lower color change. (9, 39) The high impact heat-polymerized acrylic resin with additive titanium oxide nanofillers exhibited the greater color change than the conventional heat-polymerized and microwave-polymerized acrylic resins. (76) However, the acrylic resin with the additive zirconium nanoparticles showed significant low color change. (34)

5. The usage of denture cleansers – Some studies reported the effect of color change on the immersion of the different denture cleansers. The longer immersion was, the greater color change of the acrylic resin. (9, 63, 64, 77)

Moreover, the studies reported the different color change in each acrylic resins might be the effect from surface roughness of the materials which the standard recommendation of Ra for defensing the biofilm accumulation on the surface of acrylic denture base resin was less than 30-750 nm. (78, 79)

Nanohardness and elastic modulus of acrylic resins

Hardness is one of the considered mechanical properties which indicates the resistance to the plastic deformation of the surface penetration indented from a hard asperity with non-critical force. The low hardness indicates the soft material. (7, 18) Several hardness testing methods are available depending on the shape, the size and the materials of the indenters used to deform the surface. The simple, reproducible, cheap and nondestructive hardness tests method could be classified to be macro-indentation test with the load over 1 kg and micro-indentation test with load less than 1 kg. (80, 81)

Macrohardness testing

1. Brinell hardness testing is the oldest hardness testing method especially for metals and alloy in dentistry. Basically, a 1.6 mm in diameter small steel or tungsten carbide ball is loaded with the weight of 123 N on the testing object for 30 seconds. The Brinell hardness number (BHN) is computed as a ratio of the load applied to the produced area of the indentation (kg/mm^2). The harder materials show the high BNH with the small area of indentation. The Brinell hardness test yields relatively large indentation area; therefore, the object with concrete hardness would not be suitable for this method.

2. Rockwell hardness testing is a rapid hardness testing method which a 1.5 – 13 mm diameter ball diamond indenter with the minimum load of 10 N and the major load of 60, 100 and 150 N is applied on the testing object surface. This method is suitable for the viscoelastic materials and the plastics because of the remaining loading time selected for measuring the percent of recovery. (81)

Apart from the macrohardness test with high load and larger indentation area, several materials with microstructural constituents and microphases which need more accuracy of the measurement required the hardness testing machine with smaller indenter size and controllable location. Therefore, the microindentation hardness testing machine were invented. (80)

Microhardness testing

1. Vickers hardness testing is a microindentation testing method with a 136-degree diamond square-based pyramidal indenter which is suitable to measure very hard materials and creates a small indenting area. The diagonal shaped impression ranging 10 – 100 μm is created under the applied load of 1 N or less, and then calculated the hardness value followed eq 2. (80-82)

$$\text{Hardness Value} = \frac{KP}{L^2} \qquad \text{(eq 2)}$$

where K is equal to 1.854, P the selected of applied loads and L the average diagonal length of the impression.

2. Knoop hardness testing needs a diamond rhombic-based pyramidal indenter in cooperated with the load of 1 N or less. The impression of the Knoop indenters presents the diagonal shape with a short- and a long-diagonal lines, and the ratio of the long line to the short line is 7:1. The Knoop hardness number (HK) is also calculated following the eq 2; however, L is the longest diagonal line, P the applied loads and K the constant number of 14.229. (81, 83)

Nanohardness testing

The nanoindentation method is a hardness measurement in much smaller scale. The testing method could measure the hardness and the elastic modulus of the materials while the microindentation can only perform the hardness measurement. (84) The nanohardness testing machine could calculate both the nanohardness and the elastic modulus from the curve of penetrated depth when the load was applied. A pyramidal diamond Berkovich indenter with triangular tip at the bottom in combination with the load of 0.1 – 100 mN (±1 μN) applies to the object surface, and then creates an impression depth of less than 50 nm or equivalent (±0.2 nm). (80, 84, 85)

The depth-sensing equipment in the nanohardness testing machine can continuously measure the hardness without the visualization of the impression as the microindentation testing machine but the correlation between the microhardness and the nanohardness in many materials was still unclear because of the different load and morphology of the indenter. (82, 86) In corporation with a computer-controlled high precision X-Y table, the nanohardness and elastic modulus was calculated from the load-displacement graph as shown in Figure 3. The maximum indentation load (P_{max}), the indenter displacement at the maximum load (h_{max}), the displacement at the final depth of the contact impression after unloading when the plastic depth is defined to calculate the area from the depth of the indenter in contact with the sample under load (h_f) and the initial unloading stiffness from the slope of the linear which fit tangent to the unloading curve at maximum load (S) are the factors observed. (80, 81)

The nanohardness number is calculated from the load-displacement graph by the proportion of the maximum applying load (P) and the surface area at maximum applying load (A) following eq 3. (80, 84)

$$\text{Nanohardness} = \frac{P}{A} = \frac{P}{k\,(h_p)^2} \qquad \text{(eq 3)}$$

where k is 1.167 as the geometric constant of indenter from Berkovich tip and h_p; the depth of the pressing area that is permanent deformation

Elastic modulus

The elastic modulus represents the elastic deformation of the materials when the gradually increasing force was applied. The measurement of elastic modulus could be measured from the direct methods, for example flexural strength test and the tensile strength test, and the indirect method as the depth-sensing nanoindentation test. The common flexural strength test could be performed by the three-point bending method that the compressive load would apply on the tested specimen using a universal testing machine until failure, and then calculate the elastic modulus from the linear slope of the stress-strain curve. (Figure 4) The elastic modulus of the flexural strength test is the results of the compressive strength at the loading site and the tensile strength at the opposite site. Therefore, the tensile strength test is another measurement of the elastic modulus that the load would apply on two ends of the specimen apart. The elastic modulus of the tensile strength test was calculated tensile modulus following eq 4. (18, 87)

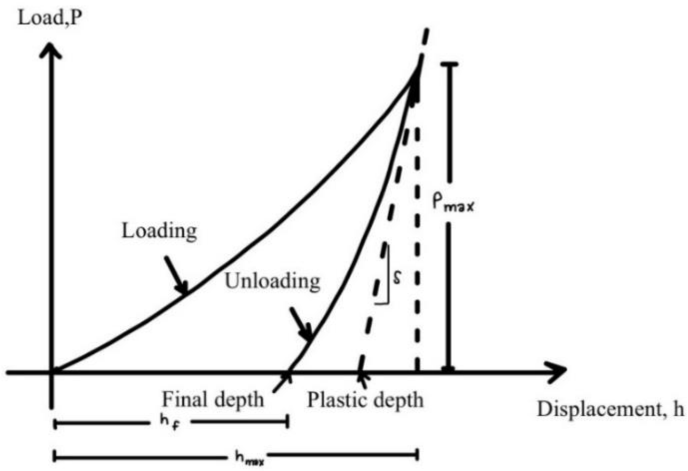


Figure 3 Load-displacement curve of nanoindentation test

Modified from (1) Willems G, Celis JP, Lambrechts P, Braem M, Vanherle G. Hardness and Young's modulus determined by nanoindentation technique of filler particles of dental restorative materials compared with human enamel. J Biomed Mater Res 1993;27:747-55., (2) Suwannaroop P, Chaijareenont P, Koottathape N, Takahashi H, Arksornnukit M. In vitro wear resistance, hardness and elastic modulus of artificial denture teeth. Dent Mater J 2011;30:461-8.

While, the correlation between the flexural modulus from conventional method and the Vicker's hardness test were confirmed, (88) the correlation between nanohardness and elastic modulus from the depth-sensing nanoindentation machine in several resin composites were also reported. (89) Moreover, the elastic modulus of the materials from the nanohardness testing machine could be measured without the specimen failure comparing the direct methods and calculated at maximum loading force (E) as followed eq 4. (80, 84)

$$E = \frac{\text{Stress}}{\text{Strain}} = \frac{\sigma}{\varepsilon} = \frac{P/A}{\Delta I/I} \qquad \text{(eq 4)}$$

where P is the applied force, A; the cross-sectional area of the material under stress, ΔI; the increase length and I; the original length.

$$\frac{1}{E^*} = \frac{1-v^2}{E} + \frac{1-v_i^{\,2}}{E_i} \qquad \text{(eq 5)}$$

where E_i is the elastic modulus of testing specimen, E^*; the reduced modulus calculated from the slope of regression line at maximum loading force of unloading curve of the load-displacement graph (Figure 3), E; the elastic modulus of Berkovich diamond indenter as 1,050 GPa, v; the Poisson's ratio of Berkovich indenter as 0.07 and v_i; the Poisson's ratio of acrylic resin as 0.38. (84)

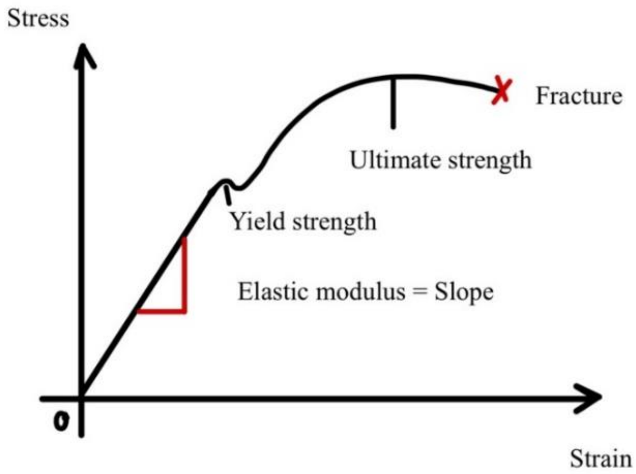


Figure 4 Stress-strain curve

Modified from Lee HH, Lee CJ, Asaoka K, Correlation in the mechanical properties of acrylic denture base resins. Dent Mater J 2012;31:157-64.

The factors related to the hardness and the elastic modulus

The hardness and the elastic modulus of the acrylic resin denture base material could predict the survival of the removable prostheses. Several factors effect on these mechanical properties including polymerization methods (15, 16, 19-21, 42, 44, 46, 51, 52, 90), the composition of each type of denture base resins and other additional compositions (17, 21, 22, 36, 37), immersion in the disinfectant solutions (77, 91, 92), the testing velocity, the various loads to the specimens and the distance between each tested impression (37, 80, 84), the pH of the storage solution and the water absorption or the adsorption of the materials. (93-98)

1. Polymerization methods – Denture base acrylic resins with the different polymerization methods and the post-polymerization after activation by environmental stimuli were reported the effect on the hardness and the elastic modulus. (15, 19-21, 44, 46, 90) One study reported the greater hardness of the conventional heat-polymerized resins than the microwaved-polymerized, the auto-polymerized denture base resins. (19) The other studies reported that the hardness, the transverse strength, the flexural strength, the tensile strength and the elastic modulus of the light-polymerized denture base resins were greater than the conventional heat and self- cured denture base resin. (15, 20, 21, 44, 46) Comparing the conventional heat-cured resins, several studies reported the equal transverse strength and flexural modulus between conventional heat-polymerized resins and microwaved-polymerized resins. (16, 42, 52, 53)

2. Composition of each type of denture base resins – The structure and molecular weight of the polymer might affect on the hardness and elastic modulus. The presence of the additive spherical amorphous silica fillers of the light-polymerized denture base resin could increase the hardness and the elastic modulus comparing to the conventional heat-polymerized and self-polymerized denture base resins. (21) However, another study reported no statistical difference of the hardness between the heat-polymerized and the auto-polymerized denture base materials because the new auto-polymerized resins were changed the initiator system and replaced copper and barbituric acid ions instead of tertiary amine. (22) Moreover, the injection-molding denture base materials with mainly polyamide resins was reported the lower hardness and elastic modus than other conventional denture base resins because of their components. (17, 36, 37)

3. Immersion in disinfectant solutions – The disinfectant solutions do not only keep the prostheses cleaned, but also reduce the hardness of the acrylic resin after some period of usage. Therefore, the reduction of hardness after immersing in in the disinfectants was significantly greater in the conventional heat-polymerized resin than high impact and polyamide acrylic resins. It might be concluded that the resistance of the material to the action of solvents were increased by the cross linking of acrylic resin materials. (77, 91) One study, however, reported no significant difference of the hardness among the denture base acrylic resins after immersing in the sodium perborate or microwaved irradiation. (92)

4. Testing velocity, various loads to specimens and distance between load to specimen site – The testing condition is also a factor influencing to the hardness and the elastic modulus. The previous studies set the various testing situations. The constant speed of 0.05-0.2 mN/s, the maximum load of up to 1-10 mN and the space between two impressions of 50 µm were reported. (37, 80, 84)

5. The pH of the storage solution and Water absorption or adsorption of the materials – The polymeric based materials were reported the influence of the acidicity of the storage solution and water sorption to decrease the elastic modulus of the materials. (93, 94) Moreover, the residual monomer content in each polymerizing acrylic denture base resin combination with the water absorption or adsorption properties was decrease the mechanical properties. (95-98)

6. The polymerization shrinkage of the materials – The correlation between stress from polymerization shrinkage during polymerization and Knoop hardness in the resin composites, meanwhile the much low correlation to the flexural modulus were also reported. (99) However, the polymerization shrinkage of the denture base acrylic resin affected on other mechanical properties still have no report yet.

CHAPTER III

RESEARCH AND METHODOLOGY

The materials used

Three commercial denture base polymeric resins used in this study were shown in Table 2

Table 2 The materials used in this study and curing methods of denture base resins by manufacturers.

Types	Code	Manufacturers	Curing methods
Heat-cured acrylic resin	TRX	Triplex®, Ivoclar Vivadent, Schaan, Liechtenstein	Heat-polymerization under 80˚C for 8 hours
Microwave-cured acrylic resin	BTC	Basis TWIN CURE®, Yamahashi Dental MFG.Co., Aichi, Japan	Heat-polymerization by using microwave to the flask at 500W for 3 minutes
Light-cured acrylic resin	TRD	Triad®, Dentply International, Inc., York, Pa.	Light-polymerization by blue light (300-500 nm)

Regarding the fabrication of the heat-cured and the microwave-cured acrylic resin groups, sixty 9.0±0.1 mm in diameter and 2.0±0.1 mm in thickness disc-shaped specimens were fabricated in the plaster stone molds and flasks with the standard silicone specimens, and then the standard silicone specimens were removed before packing and curing the mixture of powder and liquid following the manufacturer recommendations. The additive nylon in the powder was removed before mixing. (Figure 5a-b) The light-cured acrylic resin specimens with the same shape and size were produced thirty specimens with the 9.0±0.1 mm inner diameter and 2.0±0.1 mm in thickness glass tubes, and then were polymerized with the double glass slide technique and the laboratory light-curing oven (blue light 500 nm,120 W / LABOLIGHT LV-III, GC corp., Japan) for 5 minutes. (Figure 5c) All specimens were polished by using the SiC abrasive papers until the #1000 grid and the suspension of alumina oxide (0.05 μm) in water before ultrasonic clean for 10 minutes. All specimens were immersed in the distilled water at 37˚C for 24 hours before test.

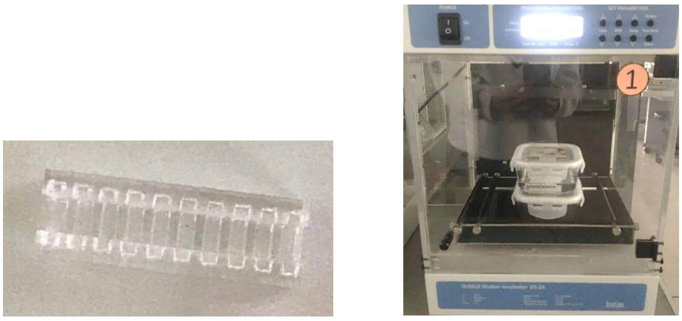
(a) (b) (c)

Figure 5 Specimen preparation of (a) the heat-cured acrylic resin group (b) the microwave-cured acrylic resin group (c) the light-cured acrylic resin group.

The color stability test

All specimens in each group were randomly divided into three interventions. The first group was immersed in the instant coffee solution (2.3 g/ 120 mL) at 37ºC which was changed every 24 hours, while the second group was immersed in the distilled water at 37ºC and then brought the two storage of setting intervention into an orbital shaker incubator (ES-20 Biosan, Medical-Biological Research & technologies, Liga, Latvia) at 37±2°C and the frequency of 1 Hz. The other was stored in the dry condition, and wrapped with foil paper at room temperature as a control group. All group were placed perpendicular and parallel from each other in the acrylic resin racks. (Figure 6)

The color of all specimens were measured by a spectrophotometry (UltraScan PRO, Hunter Associates Laboratory, Inc., Virginia, USA) before testing as the baseline data. The ΔE^*, ΔL^*, $\Delta a^*$and Δb^* were observed and calculated comparing the baseline color, following the eq 1 at day 1, and then every week for 2 months.

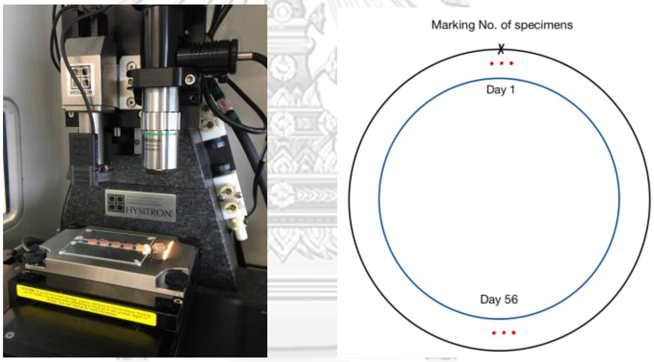


(a) (b)

Figure 6 Color stability test (a) acrylic resin rack (b) an orbital shaker incubator

The nanohardness and the elastic modulus

After the measurement of the color difference by spectrophotometry in color stability test, the nanohardness and elastic modulus were measured using a nanoindentation testing machine (Hysitron Ti Premier, Billerica, Massachusetts, USA) (Figure 7a) including a penetrating three-sided pyramidal diamond Berkovich indenter, an optical microscope and a mobile table. Three randomly chosen specimens from each experimental group were tested after 1-day and 56-day in storage media following the mapping diagram as shown in Figure 7b. Each specimen was pressed three impressions which were the 50 µm apart from each other in a linear line with the maximum load of 10 mN and a constant speed of 0.1 mN/s. The load-displacement curve was automatically generated and calculated the nanohardness and elastic modulus using the plug-in TriboScan™ software following eq 3, 5.



(a) (b)

Figure 7 The nanohardness and the elastic modulus test (a) the nanoindentation machine and (b) the testing map on the representative specimen diagram.

The color parameter, the nanohardness and the elastic modulus of all groups were statistically analyzed using the SPSS program for window version 22.0 (IBM Corp. 2013, Armonk, New York, USA). The Kolmogrov-Smirnov test were firstly proved the normal distribution, and then the Levene's test were used for indicating the homogeneity of variance. The repeated mixed analysis of variance followed by the multiple comparison (Tukey's HSD) and pair sample t-test were performed at the significant level of 95%

CHAPTER IV

RESULTS

The color stability including ΔE^*, ΔL^*, Δa^* and Δb^* of all groups after immersed in the coffee, water and kept in the dark container for 1 day, 7 day, 28 day and 56 day are illustrated in Table 5. Before the color stability examination, the surface roughness (Ra) of all specimens were confirmed for controlling the effect of the surface roughness on the color staining. The average Ra (±SD) of the TRX (12.41±1.06 nm) were the greatest, following with TRD (7.81±2.05 nm) and BTC (3.01±0.23 nm) respectively. The repeated mixed analysis of variance (Table 3) suggests significant interaction among the types of denture base materials, storage media and the storage period as well as the interaction between factors and each individual factor. ($p < 0.001$) After 56-day storage in the coffee, the color of all materials were significantly the greatest ΔE^*, darker (ΔL^*), shift to the green (Δa^*) and the yellow (Δb^*), whereas TRX immersed in the distilled water showed no significant color change during the experiment period. The ΔE^* of BTC and TRD became significant difference due to lower ΔL^* and Δa^* (darker and shift to green) at 7 day, but Δb^* were almost similar at day 56.

Table 3 Repeated mixed ANOVA of color difference (ΔE^*)

	Type III Sum of Squares	df	Mean Square	F	Sig
Material	490.207	2	245.104	202.978	<.001
Storage media	2367.207	2	1183.603	980.180	<.001
Material *Storage media	574.304	2	143.576	118.900	<.001
Error (Between subjects)	97.810	81	1.208		
Time	989.719	1.627	608.433	429.574	<.001
Time*Material	364.803	3.253	112.132	79.169	<.001
Time*Storage media	1431.553	3.253	440.026	310.673	<.001
Time*Material*Storage media	487.985	6.507	74.998	52.951	<.001
Error (Within subjects)	186.621	131.760	1.416		

The ΔE^* of all experimental groups kept in dark chamber were not significant difference until day 56 storage. ΔL^* of TRX and TRD showed darker since the day 7, but that of BTC became darker at day 56. The Δa^* and the Δb^* of TRX showed no color change, while the BTC and TRD were shift to the green at day 56. TRD was shift to the yellow at day 7.

Table 4 The ΔE^*, ΔL^*, Δa^* and Δb^* of color parameters at day 1, 7, 28, 56

		Coffee				Distilled water				Dark			
	Material	Day 1	Day 7	Day 28	Day 56	Day 1	Day 7	Day 28	Day 56	Day 1	Day 7	Day 28	Day 56
ΔE^*	TRX	0.86^{Aa} (0.01)	1.75^{Ab} (0.01)	2.09^{Ac} (0.01)	4.40^{Ad} (0.01)	0.51^{Aa} (0.03)	0.40^{Aa} (0.07)	0.76^{Aa} (0.02)	0.73^{Aa} (0.01)	0.55^{Aa} (0.08)	0.30^{Aa} (0.02)	0.43^{Aa} (0.01)	0.45^{Aa} (0.01)
	BTC	0.78^{Aa} (0.01)	4.30^{Bb} (0.01)	8.92^{Bc} (0.02)	12.52^{Bd} (0.02)	0.33^{Aa} (0.01)	0.80^{Ab} (0.03)	0.71^{Ab} (0.02)	0.71^{Ab} (0.05)	0.36^{Aa} (0.09)	0.57^{Ba} (0.02)	0.42^{Aa} (0.01)	0.57^{Aa} (0.01)
	TRD	0.95^{Aa} (0.03)	4.33^{Bb} (0.02)	11.99^{Cc} (0.02)	20.81^{Cd} (0.02)	0.40^{Aa} (0.02)	1.45^{Bb} (0.02)	3.44^{Bc} (0.01)	3.25^{Bc} (0.01)	0.41^{Aa} (0.01)	0.48^{ABa} (0.01)	0.38^{Aa} (0.01)	0.42^{Aa} (0.02)
ΔL^*	TRX	-0.72^{Aa} (0.01)	-1.34^{Aa} (0.01)	-2.24^{Ab} (0.01)	-4.00^{Ac} (0.01)	-0.18^{Aab} (0.11)	0.01^{Abc} (0.07)	0.52^{Ac} (0.01)	-0.62^{Aa} (0.01)	0.44^{Aa} (0.09)	0.01^{Ab} (0.03)	0.28^{Aab} (0.01)	-0.40^{ABc} (0.01)
	BTC	-0.43^{Ba} (0.02)	-2.34^{Ba} (0.03)	-5.91^{Bb} (0.01)	-9.32^{Bc} (0.02)	-0.17^{Aa} (0.01)	-0.68^{Bb} (0.03)	-0.59^{Bb} (0.01)	-0.57^{Ab} (0.05)	-0.14^{Ba} (0.10)	-0.31^{ABa} (0.01)	-0.29^{Ba} (0.01)	-0.68^{Ab} (0.01)
	TRD	-0.42^{Ba} (0.01)	-2.86^{Cb} (0.01)	-9.37^{Cc} (0.01)	-17.56^{Cd} (0.02)	0.08^{Aa} (0.01)	-0.94^{Bb} (0.01)	-3.18^{Cc} (0.01)	-3.20^{Bc} (0.01)	0.11^{Ba} (0.01)	-0.41^{Bb} (0.01)	-0.24^{Bb} (0.01)	-0.22^{Bb} (0.01)
Δa^*	TRX	-0.24^{Aa} (0.02)	-0.74^{Ab} (0.01)	-0.99^{Ab} (0.01)	-1.67^{Ac} (0.02)	-0.12^{Aa} (0.09)	0.02^{Aa} (0.05)	0.21^{Ab} (0.01)	-0.10^{Aa} (0.01)	-0.12^{Aa} (0.02)	-0.05^{ABa} (0.01)	-0.02^{Aa} (0.02)	-0.12^{ABa} (0.01)
	BTC	-0.32^{Aa} (0.02)	-1.74^{Bb} (0.03)	-2.61^{Bc} (0.03)	-3.63^{Bd} (0.02)	0.12^{Ba} (0.02)	-0.29^{Bb} (0.08)	-0.29^{Bb} (0.02)	-0.27^{Ab} (0.02)	0.05^{Aa} (0.02)	-0.13^{Aab} (0.02)	-0.05^{Aa} (0.01)	-0.22^{Ab} (0.02)
	TRD	-0.45^{Aa} (0.02)	-1.77^{Bb} (0.02)	-3.89^{Cc} (0.02)	-5.51^{Cd} (0.02)	0.25^{Ba} (0.02)	-0.28^{Bb} (0.01)	-0.60^{Cc} (0.02)	-0.29^{Abc} (0.02)	0.29^{Ba} (0.02)	-0.11^{Bab} (0.02)	0.12^{Aab} (0.02)	0.04^{Bb} (0.01)
Δb^*	TRX	0.35^{Aa} (0.02)	0.72^{Aa} (0.02)	1.51^{Ab} (0.01)	0.36^{Aa} (0.03)	-0.11^{Aa} (0.06)	0.01^{Aa} (0.02)	0.27^{Ab} (0.04)	0.03^{Aab} (0.02)	-0.03^{Aa} (0.04)	-0.08^{ABa} (0.02)	-0.04^{Aa} (0.01)	-0.06^{Aa} (0.02)
	BTC	0.43^{Aa} (0.02)	3.04^{Bb} (0.01)	6.06^{Bc} (0.02)	7.33^{Bc} (0.04)	0.02^{Aa} (0.02)	-0.18^{Bb} (0.02)	-0.13^{Bb} (0.02)	0.11^{Aa} (0.02)	0.09^{ABa} (0.01)	-0.05^{Aa} (0.01)	-0.01^{Aa} (0.01)	0.08^{Aa} (0.01)
	TRD	0.67^{Ba} (0.03)	2.70^{Bb} (0.03)	6.36^{Bc} (0.03)	9.55^{Cd} (0.03)	-0.09^{Aa} (0.02)	-1.06^{Cb} (0.01)	-1.17^{Cb} (0.02)	-0.10^{Aa} (0.02)	0.11^{Ba} (0.02)	-0.17^{Bb} (0.02)	-0.02^{Aab} (0.02)	0.07^{Aa} (0.02)

The different upper-case letters in the same column show the significant level of color parameter among each material. (α = 0.05)
The different lower-case letters in the same row show the significant level of color parameter in each storage media. (α = 0.05)

Apart to the materials with the different polymerization methods, TRX showed the lowest color change and the greater darkness following BTC, while TRD showed the greatest color change after immersed in the coffee. The color of TRD became shift to greenish and yellowish rather than BTC and TRX respectively. The results were quite different when storing in the distilled water.

The color change prominently showed in the TRD, especially ΔE* and ΔL*. All specimens stored in the dark chamber showed no significant color change until day 56. Each material demonstrated the different effect of the color parameters individually. (Table 4)

Table 5 Repeated mixed ANOVA of the nanohardness (H) and the elastic modulus (E_i)

		Type III Sum of Squares	df	Mean Square	F	Sig
Nanohardness	**Material**	0.148	2	0.074	105.789	<.001
	Storage media	0.112	2	0.056	80.005	<.001
	Material*Storage media	0.007	4	0.002	2.663	0.039
	Error (Within between)	0.05	72	0.001		
	Time	0.187	1	0.187	356.218	<.001
	Time*Material	0.011	2	0.006	10.703	<.001
	Time*Storage media	0.103	2	0.052	98.238	<.001
	Time*Material*Storage media	0.004	4	0.001	2.001	0.103
	Error (Within subjects)	0.038	72	0.001		
Elastic modulus	**Material**	18.101	2	9.05	38.436	<.001
	Storage media	20.86	2	10.43	44.294	<.001
	Material*Storage media	1.053	4	0.263	1.118	0.355
	Error (Between subjects)	16.954	72	0.235		
	Time	67.284	1	67.284	275.222	<.001
	Time*Material	1.924	2	0.962	3.936	0.024
	Time*Storage media	22.190	2	11.095	45.384	<.001
	Time*Material*Storage media	1.762	4	0.440	1.801	0.138
	Error (Within subjects)	17.602	72	0.244		

The repeated mixed ANOVA of the nanohardness of the experimental groups suggested the significant differences of the interaction between time and storage media, time and type of material and type of material dand storage media including each individual influencing factors; however, the interaction among time, type of material and storage media showed no significant difference. (Table 5) The nanohardness and the elastic modulus of the tested groups at 1-day and 56-day immersion were shown in Table 6. After storage in each condition for 1 day, the nanohardness of BTC and TRD showed almost similar (0.24 – 0.25 GPa), accepting for TRD in coffee (0.22 GPa). TRX showed the lowest nanohardness (0.15 – 0.17 GPa). The nanohardness of all groups immersed in coffee and distilled water decreased, but those stored in the dark chamber showed almost similar after 56 days. TRD seemed to decrease more than the others.

Table 6 The means (Stardard deviations, SD) of the nanohardness (H) and the elastic modulus (E_i) at day 1, 56

		Coffee		Distilled water		Dark	
	Material	Day 1	Day 56	Day 1	Day 56	Day 1	Day 56
Nanohardness (GPa)	TRX	0.17^{Aa} (0.00)	0.06^{Ab} (0.01)	0.15^{Aa} (0.02)	0.11^{Ab} (0.04)	0.17^{Aa} (0.01)	0.18^{Aa} (0.02)
	BTC	0.25^{Ba} (0.01)	0.10^{Ab} (0.03)	0.24^{Ba} (0.01)	0.19^{Bb} (0.02)	0.24^{Ba} (0.01)	0.24^{Ba} (0.01)
	TRD	0.22^{Ca} (0.02)	0.08^{Ab} (0.05)	0.24^{Ba} (0.02)	0.16^{Bb} (0.03)	0.25^{Ba} (0.04)	0.21^{Ca} (0.03)
Elastic modulus (GPa)	TRX	2.98^{Aa} (0.14)	0.82^{Ab} (0.29)	2.68^{Aa} (0.55)	1.49^{Ab} (0.89)	3.04^{Aa} (0.12)	2.51^{Ab} (0.54)
	BTC	3.65^{Ba} (0.31)	1.28^{Ab} (0.80)	3.37^{Ba} (0.10)	2.68^{Bb} (0.22)	3.34^{ABa} (0.16)	3.34^{Ba} (0.22)
	TRD	3.62^{Ba} (0.34)	1.40^{Ab} (0.88)	3.87^{Ca} (0.41)	2.23^{Bb} (0.35)	3.80^{Ba} (0.66)	2.99^{ABa} (0.57)

The different upper-case letters in the same column show the significant level of color parameter among each material. (α = 0.05), The different lower-case letters in the same row show the significant level of color parameter in each storage media. (α = 0.05)

The ANOVA of the elastic modulus also suggested the same fashion as the nanohardness, accepting the interaction between material and storage media. The elastic modulus of BTC and TRD were almost similar, and greater than TRX. Only TRX and BTC in dark chamber showed significantly different in day 1. The elastic modulus of all groups after immersed in coffee and water decreased after 56 day. BTC and TRD in dark condition showed no significant difference between 1-day and 56-day storage.

CHAPTER V
DISCUSSION AND CONCLUSION

Discussion

Regarding the color stability of the denture based acrylic resins, the repeated mixed analysis of variance rejected the null hypothesis which confirmed the effect of the immersion time, the type of materials with different polymerization methods and storage media over the storage periods on the color change, also the interactions between factors and each individual factor. ($p < 0.001$) Similar to the present study, the effect of the interaction among the acrylic resins with different curing methods, the beverage used over the immersion time, and each considered factors on the color changes were confirmed. (9, 65) Another study also reported the significant effect of the polymerization method of the acrylic resins and the beverage on the color stability (69). The result would be explained by the remaining residual monomer amount of the acrylic resins with different polymerization methods and the environmental condition during the polymerization that affected on the amount of leached out monomer from the resin surface or the deeper layer. (66, 67) This phenomenon might increase the absorption or adsorption of colorants into the denture base acrylic resins. An *in vitro* study of the in-laboratory fabricated resin composites with different degree of conversion on the color change after stored in water reported that the resin composite with greater degree of conversion demonstrated the lower internal color change from the lower existing double bond carbon groups. (68) Moreover, another study (65) showed the greater color change after simulated material degradation using an ultraviolet B radiation machine than the control group without the UVB accelerated.

The effect of beverage on ΔE^* of the denture base acrylic resins were confirmed similar to the other studies. (12, 61, 69-72) The color of the groups immersing in coffee increased greater than that of the groups immersing in distilled water and storing in the dark chamber. Considering all groups immersed in the coffee solution, the ΔE^* of all materials significantly increased in day 7. (70, 71) TRD showed the greater color stability than the others. The explanation would be the difference of the composition and the polymerization methods. Although TRX with heat-polymerizing method and BTC with either selective heat shock method and microwave-polymerizing method consist of the almost similar formula, mainly PMMA and MMA, the clue of the greater color change of BTC than that of TRX would be the difference of the polymerization method. (9, 63) However, TRD which is consist of UDMA, some additive organic fillers and tertiary amine, and polymerized by the blue light showed the greatest color change. The present of UDMA would be under the influence of water sorption, and consequently degraded and leached out of the UDMA. (61) Apart to the ΔL* Δa* and Δb*, all specimens immersed in coffee became darker, less red and more yellow (12, 61, 69-72), but Foungfu and Koottathape (70, 71) showed the acrylic resin denture teeth darker, redder and yellower. That might be the color baseline of the denture teeth acrylic resins which display the more yellow and less red comparing to the color baseline of the denture base acrylic resin. Moreover, the presence of several color pigment molecule, mainly tannin with the brown and the compound pigments of chlorogenic acid which later decomposed to be the greenish pigment and tannic pigments would turn the specimen color to be darker and greener after roasting, blending and brewing. (73) Similar to our previous study (70, 71), the yellow occurred from another pigment of derivative ionic phenolic compound molecules in coffee. (61, 72)

Considering all groups immersed in the distilled water, TRD group showed the greater ΔE* than BTC and TRX since day 7. The reason would be the exposure of the amorphous silica fillers on TRD surface after immersion. (21) Moreover, the formation of the hydrophilic functional UDMA in TRD might increase the water absorption properties rather than the linear chain polymer in BTC and TRX. (95)

Nevertheless, the water sorption and the polymerization methods including the heat- and microwave-polymerization were not correlated. (96) The tested material kept in the dark chamber were included in this study with the purpose of the effect of the remaining monomer on the color change. The acrylic resins after the polymerization following the manufacturer recommendation theoretically produced the residual monomer on the material surface which might increase the color change of acrylic resins. Comparing to the experimental groups in water, the ΔE* of the groups kept in the dark chamber demonstrated no significant difference during the experiment periods;

therefore, the post-cured degree of conversion and the residual monomer amount on the surface of denture base resin material might be a crucial factor affecting on the color stability.

The surface roughness would be considered as a factor influencing on the color stability. (78) The present study measured the surface roughness of all specimens for eliminating this confounding factor. The ISO standard (8) suggested only the polishing until the 1,000-grid SiC abrasive paper following the alumina oxide paste; however, the present study fabricated the specimens with Ra of 3.01 – 13.41 nm which meet the standard, and smoother than the clinical recommendation (less than 30 – 750 nm) for preventing the biofilm accumulation on the removable prostheses surface. (79) The TRD showed the greatest ΔE^* although the surface roughness met the acceptable roughness. That could be explained by the preparation from the manufacturer of TRD in the plastic sheet form and the fabrication procedure that different from TRX and BTC under the hydraulic pressure of 2 bars.

Comparing to the patient behavior, the average number of coffee which is consumed daily estimated to be 3.2 cups a day with 120 ml cup. (74) The one-day immersion in the coffee solution in this *in vitro* study would equal to approximately 3 months of coffee drinker, and 56-day immersion equal to 14.7 years. However, the storage time in this *in vitro* study was designed to cover the average service time of the prosthesis wear, also the factor of the daily cleaning protocol was not considered. Apart to the perception of the color change, the 50% of normal people could distinguish two colors with the ΔE^* greater than 1.5, and could not clinically accept the color difference greater than 3.7. (60, 62) The present results showed the ΔE^* of TRX greater than 1.5 at day 7, and those of BTC and TRD greater than 1.5 before day 7. The color change of BTC and TRD were greater than the acceptable color change at day 7, while those of TRX were at day 56.

Considering the mechanical properties of the materials after stored in the different conditions, the nanohardness of BTC and TRD were greater than TRX. The similar result pointed out that the transverse strength and microhardness of the light-polymerizing acrylic resin were significantly greater than conventional heat-polymerizing acrylic resin, and emphasized the presence of the amorphous silica filler with highly cross-linked UDMA matrix which would increase microhardness and flexural modulus, but would reduce fracture toughness and impact strength. (20) Although TRX and BTC were almost similar composition containing mainly PMMA with different polymerization method, the nanohardness of BTC were greater than that of TRX at 1 day storage in coffee and water. The similar study of the nanohardness study between the other heat-polymerizing and the microwave-polymerizing acrylic resins confirmed the present study results. (85) However, the Vicker's hardness study of the same acrylic resin with microwave activation and heat activation presented no significant difference between the polymerization method. (54) The correlation between the microhardness and the nanohardness was still elucidated because several factors, i.e. the morphology of the indenter, the mechanical properties of the tested material, were significantly affected on the results. (82) Furthermore, the nanohardness of the groups immersed in coffee and distilled water decreased, excepting the groups stored in the dark chamber. The polymerization reaction of polymer generally keep continuing after the activation with the environmental stimuli such as the light, environmental temperature etc.; therefore, the nanohardness and elastic modulus of polymer should be greater after polymerization for a period of time. (90) The present results contrary decreased after 56-day storage especially in coffee and water that might be the effect of free radical monomer leach out from the specimen surface in the combination with the water adsorption and absorption upon the material properties. (95-98)

The elastic modulus could be determined by the several conventional mechanical testing methods such as the flexural strength test, tensile strength test, and the indirectly calculated by the correlation between surface hardness and the elastic modulus following eq 3 and eq 5. The correlation between the nanohardness and the elastic modulus of several resin composites were reported as a nonlinear regression with the contingency of determinant of 0.88. (89) The present research considered the latter method because this study method needed to examine both color stability and the mechanical properties in the same specimen at the different period. The elastic modulus of TRD were similar to BTC, and greater than TRX at 1-day storage in coffee and distilled water, and then those of all specimens dramatically decreased at 56-day storage. The decrease of the elastic modulus might be under the influence of the water sorption of the hydrophilic material with high polarity, especially in the polymeric based materials. (93) Moreover, the elastic modulus of the specimens immersed in coffee seemed to be reduced much more than those in distilled water.

That might be the reason of the acidity of coffee approximately 5.5-6.0 (73), and then the polymeric material would be decomposed in the acidic beverage, and reduced the mechanical properties including the elastic modulus. (94) This study included all materials stored in the dark chamber because the remaining monomer might not only be a crucial factor that affected on the color stability, but also the nanohardness and the elastic modulus. (98) This study presented no significant difference of elastic modulus in BTC and TRD in dark chamber between 1-day and 56-day storage, whereas that of TRX significantly reduced at 56-day storage. One of the importance factors affecting on the color stability as well as the nanohardness and the elastic modulus would be the voids, the grooves or the pits on the material surface. One representative specimen of TRX in the dark chamber which was examined both the color stability and the mechanical properties showed the decrease of the elastic modulus after 56-day storage rather than the other specimens in the same group due to the comparable presence of voids and scratches shown in Figure 8.

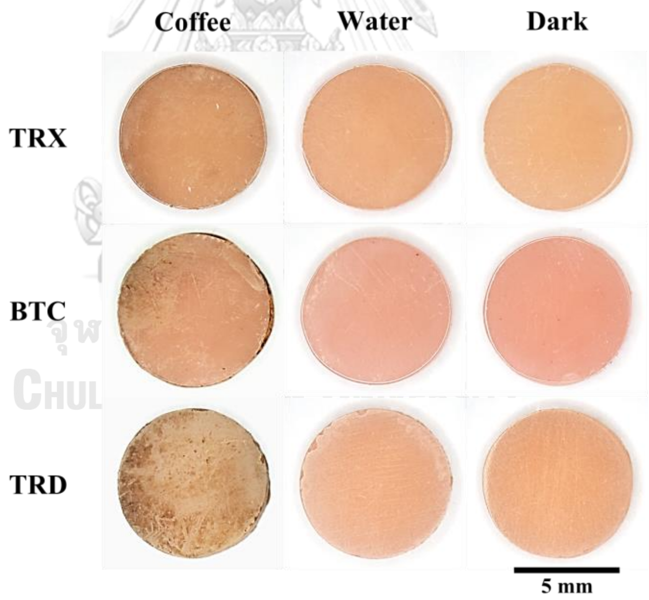


Figure 8 All specimens after 56-day storage in coffee, distilled water and kept in dark chamber.

The reliability and the correlation between the nanohardness and the microhardness are also considerably. Several surface hardness studies of the polymeric materials have been considered the microhardness indentor, for example Vicker's hardness (20) and Knoop's hardness (83), while the others considered the nanohardness indentor (Berkovich diamond indenter). The correlation between both surface hardness measurement among different surface hardness materials including metals, ceramics, rubbers and acrylic resin has been reported (82); however, a resin composite study reported no correlation between the Vicker's hardness and the nanohardness, and the Vicker's hardness showed greater than the nanohardness. (86) Aside the correlation between the surface hardness and elastic modulus, the linear correlation between the two different surface hardness tests of the resin composite was reported with the contingency of determinant of 0.99 (83), while the correlation between nanoindentation and elastic modulus of several resin composites were reported as a nonlinear regression with the contingency of determinant of 0.88. (89) The directly flexural modulus of resin composites also was correlated to the elastic modulus determined by the Vicker's hardness test with the pearson correlation of 0.94. (88) Therefore, the nanohardness and the elastic modulus determination of this study would be a reliable alternative measurement.

Clinically, the appearance, the durability and the longevity of the removable prostheses are the crucial considering factors to predict the success of the tooth/teeth replacement treatment. This *in vitro* study confirmed the influencing factors of the polymerization methods, the storage conditions on the color stability, the nanohardness and the elastic modulus of the acrylic resins. The oral cavity is considerably tremendous environment with for example the dynamic pH and oral temperature changes from the daily food and beverage, also the subcritical continuous biting load, and the daily cleaning with the disinfectant. (9, 63, 64, 77, 90, 91) The comparative report between the *in vitro* study of color stability and the predictable *in vivo* study has also not been elucidated. Moreover, the color change and the mechanical properties would be under the influence of the remaining residual monomer inside or on the acrylic resin surface, their degree of conversion and the stress from the polymerization shrinkage of the resin materials. Several studies indicated the influence of them on the color stability and the mechanical properties, and be confirmed by the present results. However, the correlation between the color stability and the nanohardness, and the elastic modulus needs to be further studied.

Conclusion

Within the limitations of the present study, the color stability, the nanohardness and the elastic modulus of the denture base acrylic resins were under the influence of the polymerization methods, the storage condition and time. All specimens stored in coffee demonstrated the greatest color change than distilled water and dark chamber after 7-day storage. TRD showed more color change than TRX and BTC in both coffee and distilled water, whereas the materials in dark chamber showed no significant difference. All materials in the coffee were significantly darker ($-\Delta L^*$), shifted to green ($-\Delta a^*$) and yellow ($+\Delta b^*$) after 56-day storage. The TRX showed no significant color change in distilled water, while the ΔL^* and Δa^* of BTC and TRD were lower (darker and shift toward green) at 7-day.

The nanohardness and the elastic modulus of all groups in coffee and distilled water decreased after 56-day storage. Those values in coffee decreased greater than those in water. TRX showed the lowest nanohardness and elastic modulus.

APPENDIX

Repeated mixed Analysis of variance: ΔE^*

Within-Subjects Factors

Measure: dE

Immertion_Time	Dependent Variable
1	dE_day1
2	dE_day7
3	dE_day28
4	dE_day56

Between-Subjects Factors

		N
Testing_Groups	BTC/C	10
	BTC/D	10
	BTC/W	10
	TRD/C	10
	TRD/D	10
	TRD/W	10
	TRX/C	10
	TRX/D	10
	TRX/W	10

Box's Test of Equality of Covariance Matrices[a]

Box's M	447.425
F	4.547
df1	80
df2	7963.845
Sig.	.000

Tests the null hypothesis that the observed covariance matrices of the dependent variables are equal across groups.

a. Design: Intercept + Testing_Groups Within Subjects Design: Immertion_Time

Descriptive Statistics

	Testing_Groups	Mean	Std. Deviation	N
dE_day1	BTC/C	.78100	.207817	10
	BTC/D	.35833	.131283	10
	BTC/W	.32700	.126934	10
	TRD/C	.95100	.298569	10
	TRD/D	.40933	.150733	10
	TRD/W	.40000	.106875	10
	TRX/C	.86000	.163775	10
	TRX/D	.54533	.268007	10
	TRX/W	.51000	.335890	10
	Total	.57133	.301066	90
dE_day7	BTC/C	4.29600	.344616	10
	BTC/D	.57400	.215021	10
	BTC/W	.79900	.367104	10
	TRD/C	4.32800	.648088	10
	TRD/D	.48367	.267930	10
	TRD/W	1.45400	.247799	10
	TRX/C	1.74600	.171672	10
	TRX/D	.29633	.131820	10
	TRX/W	.39600	.252507	10
	Total	1.59700	1.561827	90
dE_day28	BTC/C	8.92200	1.829121	10
	BTC/D	.41833	.216083	10
	BTC/W	.70900	.293767	10
	TRD/C	11.99600	1.807166	10
	TRD/D	.38267	.146673	10
	TRD/W	3.44300	.268413	10
	TRX/C	2.91000	.684673	10
	TRX/D	.42833	.283768	10
	TRX/W	.75900	.306973	10
	Total	3.32981	4.137477	90
dE_day56	BTC/C	12.51600	4.014909	10
	BTC/D	.92067	.267585	10
	BTC/W	.70800	.404567	10
	TRD/C	20.80833	2.116251	10
	TRD/D	.42333	.128438	10
	TRD/W	3.24900	.460121	10
	TRX/C	4.39900	1.190768	10
	TRX/D	.45267	.188829	10
	TRX/W	.72600	.481543	10
	Total	4.91144	6.911771	90

Multivariate Tests[a]

Effect		Value	F	Hypothesis df	Error df	Sig.	Partial Eta Squared
Immertion_Time	Pillai's Trace	.957	582.677[b]	3.000	79.000	.000	.957
	Wilks' Lambda	.043	582.677[b]	3.000	79.000	.000	.957
	Hotelling's Trace	22.127	582.677[b]	3.000	79.000	.000	.957
	Roy's Largest Root	22.127	582.677[b]	3.000	79.000	.000	.957
Immertion_Time * Testing_Groups	Pillai's Trace	1.669	12.688	24.000	243.000	.000	.556
	Wilks' Lambda	.009	39.156	24.000	229.725	.000	.793
	Hotelling's Trace	43.423	140.521	24.000	233.000	.000	.935
	Roy's Largest Root	42.011	425.361[c]	8.000	81.000	.000	.977

a. Design: Intercept + Testing_Groups
Within Subjects Design: Immertion_Time

b. Exact statistic

c. The statistic is an upper bound on F that yields a lower bound on the significance level.

Mauchly's Test of Sphericity[a]

Measure: dE

Within Subjects Effect	Mauchly's W	Approx. Chi-Square	df	Sig.	Epsilon[b] Greenhouse-Geisser	Huynh-Feldt	Lower-bound
Immertion_Time	.107	178.467	5	.000	.542	.606	.333

Tests the null hypothesis that the error covariance matrix of the orthonormalized transformed dependent variables is proportional to an identity matrix.

a. Design: Intercept + Testing_Groups
Within Subjects Design: Immertion_Time

b. May be used to adjust the degrees of freedom for the averaged tests of significance. Corrected tests are displayed in the Tests of Within-Subjects Effects table.

Tests of Within-Subjects Effects

Measure: dE

Source		Type III Sum of Squares	df	Mean Square	F	Sig.	Partial Eta Squared
Immertion_Time	Sphericity Assumed	989.719	3	329.906	429.574	.000	.841
	Greenhouse-Geisser	989.719	1.627	608.433	429.574	.000	.841
	Huynh-Feldt	989.719	1.819	544.025	429.574	.000	.841
	Lower-bound	989.719	1.000	989.719	429.574	.000	.841
Immertion_Time * Testing_Groups	Sphericity Assumed	2284.342	24	95.181	123.936	.000	.924
	Greenhouse-Geisser	2284.342	13.013	175.538	123.936	.000	.924
	Huynh-Feldt	2284.342	14.554	156.956	123.936	.000	.924
	Lower-bound	2284.342	8.000	285.543	123.936	.000	.924
Error(Immertion_Time)	Sphericity Assumed	186.621	243	.768			
	Greenhouse-Geisser	186.621	131.760	1.416			
	Huynh-Feldt	186.621	147.359	1.266			
	Lower-bound	186.621	81.000	2.304			

Tests of Within-Subjects Contrasts

Measure: dE

Source	Immertion_Time	Type III Sum of Squares	df	Mean Square	F	Sig.	Partial Eta Squared
Immertion_Time	Linear	979.449	1	979.449	746.416	.000	.902
	Quadratic	6.955	1	6.955	11.206	.001	.122
	Cubic	3.315	1	3.315	8.933	.004	.099
Immertion_Time * Testing_Groups	Linear	2201.564	8	275.195	209.720	.000	.954
	Quadratic	72.692	8	9.086	14.641	.000	.591
	Cubic	10.086	8	1.261	3.397	.002	.251
Error(Immertion_Time)	Linear	106.288	81	1.312			
	Quadratic	50.271	81	.621			
	Cubic	30.062	81	.371			

Levene's Test of Equality of Error Variances[a]

	F	df1	df2	Sig.
dE_day1	2.554	8	81	.016
dE_day7	4.161	8	81	.000
dE_day28	6.820	8	81	.000
dE_day56	20.992	8	81	.000

Tests the null hypothesis that the error variance of the dependent variable is equal across groups.

a. Design: Intercept + Testing_Groups
Within Subjects Design: Immertion_Time

Tests of Between-Subjects Effects

Measure: dE

Transformed Variable: Average

Source	Type III Sum of Squares	df	Mean Square	F	Sig.	Partial Eta Squared
Intercept	2438.091	1	2438.091	2019.061	.000	.961
Testing_Groups	3431.718	8	428.965	355.239	.000	.972
Error	97.810	81	1.208			

Estimate marginal means

1. Material_Types

Measure: dE

Material_Types	Mean	Std. Error	95% Confidence Interval	
			Lower Bound	Upper Bound
heat-cured	1.169	.100	.969	1.369
Light-cured	4.027	.100	3.828	4.227
microwave-cured	2.611	.100	2.411	2.810

2. Solution

Measure: dE

Solution	Mean	Std. Error	95% Confidence Interval	
			Lower Bound	Upper Bound
coffee	6.209	.100	6.010	6.409
dark	.474	.100	.275	.674
water	1.123	.100	.924	1.323

3. Time

Measure: dE

Time	Mean	Std. Error	95% Confidence Interval	
			Lower Bound	Upper Bound
1	.571	.023	.526	.616
2	1.597	.034	1.528	1.666
3	3.330	.096	3.139	3.521
4	4.911	.168	4.578	5.245

4. Material_Types * Solution

Measure: dE

Material_Types	Solution	Mean	Std. Error	95% Confidence Interval Lower Bound	Upper Bound
heat-cured	coffee	2.479	.174	2.133	2.824
	dark	.431	.174	.085	.776
	water	.598	.174	.252	.943
Light-cured	coffee	9.521	.174	9.175	9.867
	dark	.425	.174	.079	.770
	water	2.136	.174	1.791	2.482
microwave-cured	coffee	6.629	.174	6.283	6.974
	dark	.568	.174	.222	.914
	water	.636	.174	.290	.981

6. Solution * Time

Measure: dE

Solution	Time	Mean	Std. Error	95% Confidence Interval Lower Bound	Upper Bound
coffee	1	.864	.039	.786	.942
	2	3.457	.060	3.338	3.576
	3	7.943	.166	7.611	8.274
	4	12.574	.290	11.997	13.152
dark	1	.438	.039	.360	.515
	2	.451	.060	.332	.570
	3	.410	.166	.079	.741
	4	.599	.290	.021	1.176
water	1	.412	.039	.335	.490
	2	.883	.060	.764	1.002
	3	1.637	.166	1.306	1.968
	4	1.561	.290	.983	2.139

5. Material_Types * Time

Measure: dE

Material_Types	Time	Mean	Std. Error	95% Confidence Interval Lower Bound	Upper Bound
heat-cured	1	.638	.039	.561	.716
	2	.813	.060	.694	.932
	3	1.366	.166	1.035	1.697
	4	1.859	.290	1.282	2.437
Light-cured	1	.587	.039	.509	.664
	2	2.089	.060	1.970	2.207
	3	5.274	.166	4.943	5.605
	4	8.160	.290	7.583	8.738
microwave-cured	1	.489	.039	.411	.566
	2	1.890	.060	1.771	2.009
	3	3.350	.166	3.019	3.681
	4	4.715	.290	4.137	5.292

7. Material_Types * Solution * Time

Measure: dE

Material_Types	Solution	Time	Mean	Std. Error	95% Confidence Interval Lower Bound	95% Confidence Interval Upper Bound
heat-cured	coffee	1	.860	.068	.725	.995
		2	1.746	.103	1.540	1.952
		3	2.910	.288	2.336	3.484
		4	4.399	.503	3.399	5.399
	dark	1	.545	.068	.411	.680
		2	.296	.103	.090	.502
		3	.428	.288	-.145	1.002
		4	.453	.503	-.548	1.453
	water	1	.510	.068	.375	.645
		2	.396	.103	.190	.602
		3	.759	.288	.185	1.333
		4	.726	.503	-.274	1.726
Light-cured	coffee	1	.951	.068	.816	1.086
		2	4.328	.103	4.122	4.534
		3	11.996	.288	11.422	12.570
		4	20.808	.503	19.808	21.809
	dark	1	.409	.068	.275	.544
		2	.484	.103	.278	.690
		3	.383	.288	-.191	.956
		4	.423	.503	-.577	1.424
	water	1	.400	.068	.265	.535
		2	1.454	.103	1.248	1.660
		3	3.443	.288	2.869	4.017
		4	3.249	.503	2.249	4.249
microwave-cured	coffee	1	.781	.068	.646	.916
		2	4.296	.103	4.090	4.502
		3	8.922	.288	8.348	9.496
		4	12.516	.503	11.516	13.516
	dark	1	.358	.068	.224	.493
		2	.574	.103	.368	.780
		3	.418	.288	-.155	.992
		4	.921	.503	-.080	1.921
	water	1	.327	.068	.192	.462
		2	.799	.103	.593	1.005
		3	.709	.288	.135	1.283
		4	.708	.503	-.292	1.708

One way Analysis of variance: $\Delta E^*\Delta L^*$ Δa^* and Δb^*

TRX in coffee solution

ANOVA

		Sum of Squares	df	Mean Square	F	Sig.
dE	Between Groups	70.306	3	23.435	48.246	.000
	Within Groups	17.487	36	.486		
	Total	87.793	39			
dL	Between Groups	61.033	3	20.344	50.370	.000
	Within Groups	14.540	36	.404		
	Total	75.573	39			
dA	Between Groups	10.646	3	3.549	63.979	.000
	Within Groups	1.997	36	.055		
	Total	12.643	39			
dB	Between Groups	9.005	3	3.002	11.944	.000
	Within Groups	9.047	36	.251		
	Total	18.051	39			

Multiple Comparisons

Tukey HSD

Dependent Variable	(I) Immertion_Time	(J) Immertion_Time	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
						Lower Bound	Upper Bound
dE	1.0	7.0	-.886000*	.311689	.035	-1.72545	-.04655
		28.0	-2.050000*	.311689	.000	-2.88945	-1.21055
		56.0	-3.539000*	.311689	.000	-4.37845	-2.69955
	7.0	1.0	.886000*	.311689	.035	.04655	1.72545
		28.0	-1.164000*	.311689	.003	-2.00345	-.32455
		56.0	-2.653000*	.311689	.000	-3.49245	-1.81355
	28.0	1.0	2.050000*	.311689	.000	1.21055	2.88945
		7.0	1.164000*	.311689	.003	.32455	2.00345
		56.0	-1.489000*	.311689	.000	-2.32845	-.64955
	56.0	1.0	3.539000*	.311689	.000	2.69955	4.37845
		7.0	2.653000*	.311689	.000	1.81355	3.49245
		28.0	1.489000*	.311689	.000	.64955	2.32845
dL	1.0	7.0	.615000	.284216	.153	-.15046	1.38046
		28.0	1.519000*	.284216	.000	.75354	2.28446
		56.0	3.276667*	.284216	.000	2.51121	4.04213
	7.0	1.0	-.615000	.284216	.153	-1.38046	.15046
		28.0	.904000*	.284216	.015	.13854	1.66946
		56.0	2.661667*	.284216	.000	1.89621	3.42713
	28.0	1.0	-1.519000*	.284216	.000	-2.28446	-.75354
		7.0	-.904000*	.284216	.015	-1.66946	-.13854
		56.0	1.757667*	.284216	.000	.99221	2.52313
	56.0	1.0	-3.276667*	.284216	.000	-4.04213	-2.51121
		7.0	-2.661667*	.284216	.000	-3.42713	-1.89621
		28.0	-1.757667*	.284216	.000	-2.52313	-.99221
dA	1.0	7.0	.504000*	.105326	.000	.22033	.78767
		28.0	.754000*	.105326	.000	.47033	1.03767
		56.0	1.432333*	.105326	.000	1.14867	1.71600
	7.0	1.0	-.504000*	.105326	.000	-.78767	-.22033
		28.0	.250000	.105326	.101	-.03367	.53367
		56.0	.928333*	.105326	.000	.64467	1.21200
	28.0	1.0	-.754000*	.105326	.000	-1.03767	-.47033
		7.0	-.250000	.105326	.101	-.53367	.03367
		56.0	.678333*	.105326	.000	.39467	.96200
	56.0	1.0	-1.432333*	.105326	.000	-1.71600	-1.14867
		7.0	-.928333*	.105326	.000	-1.21200	-.64467
		28.0	-.678333*	.105326	.000	-.96200	-.39467
dB	1.0	7.0	-.378000	.224188	.346	-.98179	.22579
		28.0	-1.169000*	.224188	.000	-1.77279	-.56521
		56.0	-.014000	.224188	1.000	-.61779	.58979
	7.0	1.0	.378000	.224188	.346	-.22579	.98179
		28.0	-.791000*	.224188	.006	-1.39479	-.18721
		56.0	.364000	.224188	.379	-.23979	.96779
	28.0	1.0	1.169000*	.224188	.000	.56521	1.77279
		7.0	.791000*	.224188	.006	.18721	1.39479
		56.0	1.155000*	.224188	.000	.55121	1.75879
	56.0	1.0	.014000	.224188	1.000	-.58979	.61779
		7.0	-.364000	.224188	.379	-.96779	.23979
		28.0	-1.155000*	.224188	.000	-1.75879	-.55121

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

BTC in coffee solution

ANOVA

		Sum of Squares	df	Mean Square	F	Sig.
dE	Between Groups	795.566	3	265.189	54.045	.000
	Within Groups	176.644	36	4.907		
	Total	972.210	39			
dL	Between Groups	464.765	3	154.922	49.397	.000
	Within Groups	112.906	36	3.136		
	Total	577.671	39			
dA	Between Groups	58.762	3	19.587	43.905	.000
	Within Groups	16.061	36	.446		
	Total	74.822	39			
dB	Between Groups	288.313	3	96.104	35.992	.000
	Within Groups	96.125	36	2.670		
	Total	384.438	39			

Multiple Comparisons

Tukey HSD

Dependent Variable	(I) Immertion_Time	(J) Immertion_Time	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
						Lower Bound	Upper Bound
dE	1.0	7.0	-3.515000*	.990634	.006	-6.18300	-.84700
		28.0	-8.141000*	.990634	.000	-10.80900	-5.47300
		56.0	-11.735000*	.990634	.000	-14.40300	-9.06700
	7.0	1.0	3.515000*	.990634	.006	.84700	6.18300
		28.0	-4.626000*	.990634	.000	-7.29400	-1.95800
		56.0	-8.220000*	.990634	.000	-10.88800	-5.55200
	28.0	1.0	8.141000*	.990634	.000	5.47300	10.80900
		7.0	4.626000*	.990634	.000	1.95800	7.29400
		56.0	-3.594000*	.990634	.005	-6.26200	-.92600
	56.0	1.0	11.735000*	.990634	.000	9.06700	14.40300
		7.0	8.220000*	.990634	.000	5.55200	10.88800
		28.0	3.594000*	.990634	.005	.92600	6.26200
dL	1.0	7.0	1.912000	.791995	.092	-.22102	4.04502
		28.0	5.483000*	.791995	.000	3.34998	7.61602
		56.0	8.892667*	.791995	.000	6.75964	11.02569
	7.0	1.0	-1.912000	.791995	.092	-4.04502	.22102
		28.0	3.571000*	.791995	.000	1.43798	5.70402
		56.0	6.980667*	.791995	.000	4.84764	9.11369
	28.0	1.0	-5.483000*	.791995	.000	-7.61602	-3.34998
		7.0	-3.571000*	.791995	.000	-5.70402	-1.43798
		56.0	3.409667*	.791995	.001	1.27664	5.54269
	56.0	1.0	-8.892667*	.791995	.000	-11.02569	-6.75964
		7.0	-6.980667*	.791995	.000	-9.11369	-4.84764
		28.0	-3.409667*	.791995	.001	-5.54269	-1.27664
dA	1.0	7.0	1.416000*	.298706	.000	.61152	2.22048
		28.0	2.284000*	.298706	.000	1.47952	3.08848
		56.0	3.304667*	.298706	.000	2.50018	4.10915
	7.0	1.0	-1.416000*	.298706	.000	-2.22048	-.61152
		28.0	.868000*	.298706	.030	.06352	1.67248
		56.0	1.888667*	.298706	.000	1.08418	2.69315
	28.0	1.0	-2.284000*	.298706	.000	-3.08848	-1.47952
		7.0	-.868000*	.298706	.030	-1.67248	-.06352
		56.0	1.020667*	.298706	.008	.21618	1.82515
	56.0	1.0	-3.304667*	.298706	.000	-4.10915	-2.50018
		7.0	-1.888667*	.298706	.000	-2.69315	-1.08418
		28.0	-1.020667*	.298706	.008	-1.82515	-.21618
dB	1.0	7.0	-2.613000*	.730774	.005	-4.58114	-.64486
		28.0	-5.636000*	.730774	.000	-7.60414	-3.66786
		56.0	-6.900333*	.730774	.000	-8.86847	-4.93219
	7.0	1.0	2.613000*	.730774	.005	.64486	4.58114
		28.0	-3.023000*	.730774	.001	-4.99114	-1.05486
		56.0	-4.287333*	.730774	.000	-6.25547	-2.31919
	28.0	1.0	5.636000*	.730774	.000	3.66786	7.60414
		7.0	3.023000*	.730774	.001	1.05486	4.99114
		56.0	-1.264333	.730774	.323	-3.23247	.70381
	56.0	1.0	6.900333*	.730774	.000	4.93219	8.86847
		7.0	4.287333*	.730774	.000	2.31919	6.25547
		28.0	1.264333	.730774	.323	-.70381	3.23247

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

TRD in coffee solution

ANOVA

		Sum of Squares	df	Mean Square	F	Sig.
dE	Between Groups	2926.953	3	975.651	597.730	.000
	Within Groups	75.084	46	1.632		
	Total	3002.037	49			
dL	Between Groups	2170.065	3	723.355	504.621	.000
	Within Groups	65.939	46	1.433		
	Total	2236.004	49			
dA	Between Groups	199.607	3	66.536	111.092	.000
	Within Groups	27.550	46	.599		
	Total	227.157	49			
dB	Between Groups	602.309	3	200.770	469.182	.000
	Within Groups	19.684	46	.428		
	Total	621.993	49			

Multiple Comparisons

Tukey HSD

Dependent Variable	(I) Immertion_Time	(J) Immertion_Time	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
						Lower Bound	Upper Bound
dE	1.0	7.0	-3.377000*	.494812	.000	-4.69592	-2.05808
		28.0	-11.045000*	.494812	.000	-12.36392	-9.72608
		56.0	-19.857333*	.494812	.000	-21.17625	-18.53841
	7.0	1.0	3.377000*	.494812	.000	2.05808	4.69592
		28.0	-7.668000*	.571360	.000	-9.19096	-6.14504
		56.0	-16.480333*	.571360	.000	-18.00329	-14.95738
	28.0	1.0	11.045000*	.494812	.000	9.72608	12.36392
		7.0	7.668000*	.571360	.000	6.14504	9.19096
		56.0	-8.812333*	.571360	.000	-10.33529	-7.28938
	56.0	1.0	19.857333*	.494812	.000	18.53841	21.17625
		7.0	16.480333*	.571360	.000	14.95738	18.00329
		28.0	8.812333*	.571360	.000	7.28938	10.33529
dL	1.0	7.0	2.434000*	.463702	.000	1.19800	3.67000
		28.0	8.950000*	.463702	.000	7.71400	10.18600
		56.0	17.136667*	.463702	.000	15.90067	18.37266
	7.0	1.0	-2.434000*	.463702	.000	-3.67000	-1.19800
		28.0	6.516000*	.535437	.000	5.08880	7.94320
		56.0	14.702667*	.535437	.000	13.27546	16.12987
	28.0	1.0	-8.950000*	.463702	.000	-10.18600	-7.71400
		7.0	-6.516000*	.535437	.000	-7.94320	-5.08880
		56.0	8.186667*	.535437	.000	6.75946	9.61387
	56.0	1.0	-17.136667*	.463702	.000	-18.37266	-15.90067
		7.0	-14.702667*	.535437	.000	-16.12987	-13.27546
		28.0	-8.186667*	.535437	.000	-9.61387	-6.75946
dA	1.0	7.0	1.326000*	.299731	.000	.52707	2.12493
		28.0	3.448000*	.299731	.000	2.64907	4.24693
		56.0	5.068333*	.299731	.000	4.26940	5.86726
	7.0	1.0	-1.326000*	.299731	.000	-2.12493	-.52707
		28.0	2.122000*	.346099	.000	1.19947	3.04453
		56.0	3.742333*	.346099	.000	2.81981	4.66486
	28.0	1.0	-3.448000*	.299731	.000	-4.24693	-2.64907
		7.0	-2.122000*	.346099	.000	-3.04453	-1.19947
		56.0	1.620333*	.346099	.000	.69781	2.54286
	56.0	1.0	-5.068333*	.299731	.000	-5.86726	-4.26940
		7.0	-3.742333*	.346099	.000	-4.66486	-2.81981
		28.0	-1.620333*	.346099	.000	-2.54286	-.69781
dB	1.0	7.0	-2.029000*	.253352	.000	-2.70431	-1.35369
		28.0	-5.690000*	.253352	.000	-6.36531	-5.01469
		56.0	-8.879000*	.253352	.000	-9.55431	-8.20369
	7.0	1.0	2.029000*	.253352	.000	1.35369	2.70431
		28.0	-3.661000*	.292545	.000	-4.44078	-2.88122
		56.0	-6.850000*	.292545	.000	-7.62978	-6.07022
	28.0	1.0	5.690000*	.253352	.000	5.01469	6.36531
		7.0	3.661000*	.292545	.000	2.88122	4.44078
		56.0	-3.189000*	.292545	.000	-3.96878	-2.40922
	56.0	1.0	8.879000*	.253352	.000	8.20369	9.55431
		7.0	6.850000*	.292545	.000	6.07022	7.62978
		28.0	3.189000*	.292545	.000	2.40922	3.96878

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

TRX in distilled water

ANOVA

		Sum of Squares	df	Mean Square	F	Sig.
dE	Between Groups	.909	3	.303	2.410	.083
	Within Groups	4.524	36	.126		
	Total	5.433	39			
dL	Between Groups	6.661	3	2.220	10.004	.000
	Within Groups	7.989	36	.222		
	Total	14.650	39			
dA	Between Groups	.698	3	.233	5.710	.003
	Within Groups	1.467	36	.041		
	Total	2.165	39			
dB	Between Groups	.744	3	.248	5.378	.004
	Within Groups	1.661	36	.046		
	Total	2.405	39			

Multiple Comparisons

Tukey HSD

Dependent Variable	(I) Immertion_Time	(J) Immertion_Time	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval Lower Bound	95% Confidence Interval Upper Bound
dE	1.0	7.0	.114000	.158540	.889	-.31298	.54098
		28.0	-.249000	.158540	.408	-.67598	.17798
		56.0	-.216000	.158540	.530	-.64298	.21098
	7.0	1.0	-.114000	.158540	.889	-.54098	.31298
		28.0	-.363000	.158540	.119	-.78998	.06398
		56.0	-.330000	.158540	.179	-.75698	.09698
	28.0	1.0	.249000	.158540	.408	-.17798	.67598
		7.0	.363000	.158540	.119	-.06398	.78998
		56.0	.033000	.158540	.997	-.39398	.45998
	56.0	1.0	.216000	.158540	.530	-.21098	.64298
		7.0	.330000	.158540	.179	-.09698	.75698
		28.0	-.033000	.158540	.997	-.45998	.39398
dL	1.0	7.0	-.191000	.210678	.801	-.75840	.37640
		28.0	-.702000*	.210678	.010	-1.26940	-.13460
		56.0	.435000	.210678	.184	-.13240	1.00240
	7.0	1.0	.191000	.210678	.801	-.37640	.75840
		28.0	-.511000	.210678	.090	-1.07840	.05640
		56.0	.626000*	.210678	.026	.05860	1.19340
	28.0	1.0	.702000*	.210678	.010	.13460	1.26940
		7.0	.511000	.210678	.090	-.05640	1.07840
		56.0	1.137000*	.210678	.000	.56960	1.70440
	56.0	1.0	-.435000	.210678	.184	-1.00240	.13240
		7.0	-.626000*	.210678	.026	-1.19340	-.05860
		28.0	-1.137000*	.210678	.000	-1.70440	-.56960
dA	1.0	7.0	-.132000	.090282	.470	-.37515	.11115
		28.0	-.330000*	.090282	.004	-.57315	-.08685
		56.0	-.014333	.090282	.999	-.25748	.22882
	7.0	1.0	.132000	.090282	.470	-.11115	.37515
		28.0	-.198000	.090282	.145	-.44115	.04515
		56.0	.117667	.090282	.567	-.12548	.36082
	28.0	1.0	.330000*	.090282	.004	.08685	.57315
		7.0	.198000	.090282	.145	-.04515	.44115
		56.0	.315667*	.090282	.007	.07252	.55882
	56.0	1.0	.014333	.090282	.999	-.22882	.25748
		7.0	-.117667	.090282	.567	-.36082	.12548
		28.0	-.315667*	.090282	.007	-.55882	-.07252
dB	1.0	7.0	-.118000	.096048	.613	-.37668	.14068
		28.0	-.376000*	.096048	.002	-.63468	-.11732
		56.0	-.139667	.096048	.475	-.39835	.11901
	7.0	1.0	.118000	.096048	.613	-.14068	.37668
		28.0	-.258000	.096048	.051	-.51668	.00068
		56.0	-.021667	.096048	.996	-.28035	.23701
	28.0	1.0	.376000*	.096048	.002	.11732	.63468
		7.0	.258000	.096048	.051	-.00068	.51668
		56.0	.236333	.096048	.084	-.02235	.49501
	56.0	1.0	.139667	.096048	.475	-.11901	.39835
		7.0	.021667	.096048	.996	-.23701	.28035
		28.0	-.236333	.096048	.084	-.49501	.02235

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

BTC in distilled water

ANOVA

		Sum of Squares	df	Mean Square	F	Sig.
dE	Between Groups	1.326	3	.442	4.409	.010
	Within Groups	3.608	36	.100		
	Total	4.933	39			
dL	Between Groups	1.579	3	.526	4.044	.014
	Within Groups	4.686	36	.130		
	Total	6.266	39			
dA	Between Groups	1.230	3	.410	11.866	.000
	Within Groups	1.244	36	.035		
	Total	2.473	39			
dB	Between Groups	.538	3	.179	24.464	.000
	Within Groups	.264	36	.007		
	Total	.802	39			

Multiple Comparisons

Tukey HSD

Dependent Variable	(I) Immertion_Time	(J) Immertion_Time	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
						Lower Bound	Upper Bound
dE	1.0	7.0	-.472000*	.141572	.010	-.85328	-.09072
		28.0	-.382000*	.141572	.049	-.76328	-.00072
		56.0	-.381000	.141572	.050	-.76228	.00028
	7.0	1.0	.472000*	.141572	.010	.09072	.85328
		28.0	.090000	.141572	.920	-.29128	.47128
		56.0	.091000	.141572	.917	-.29028	.47228
	28.0	1.0	.382000*	.141572	.049	.00072	.76328
		7.0	-.090000	.141572	.920	-.47128	.29128
		56.0	.001000	.141572	1.000	-.38028	.38228
	56.0	1.0	.381000	.141572	.050	-.00028	.76228
		7.0	-.091000	.141572	.917	-.47228	.29028
		28.0	-.001000	.141572	1.000	-.38228	.38028
dL	1.0	7.0	.517000*	.161356	.014	.08243	.95157
		28.0	.419000	.161356	.062	-.01557	.85357
		56.0	.409333	.161356	.071	-.02524	.84390
	7.0	1.0	-.517000*	.161356	.014	-.95157	-.08243
		28.0	-.098000	.161356	.929	-.53257	.33657
		56.0	-.107667	.161356	.909	-.54224	.32690
	28.0	1.0	-.419000	.161356	.062	-.85357	.01557
		7.0	.098000	.161356	.929	-.33657	.53257
		56.0	-.009667	.161356	1.000	-.44424	.42490
	56.0	1.0	-.409333	.161356	.071	-.84390	.02524
		7.0	.107667	.161356	.909	-.32690	.54224
		28.0	.009667	.161356	1.000	-.42490	.44424
dA	1.0	7.0	.413000*	.083122	.000	.18913	.63687
		28.0	.408000*	.083122	.000	.18413	.63187
		56.0	.392667*	.083122	.000	.16880	.61653
	7.0	1.0	-.413000*	.083122	.000	-.63687	-.18913
		28.0	-.005000	.083122	1.000	-.22887	.21887
		56.0	-.020333	.083122	.995	-.24420	.20353
	28.0	1.0	-.408000*	.083122	.000	-.63187	-.18413
		7.0	.005000	.083122	1.000	-.21887	.22887
		56.0	-.015333	.083122	.998	-.23920	.20853
	56.0	1.0	-.392667*	.083122	.000	-.61653	-.16880
		7.0	.020333	.083122	.995	-.20353	.24420
		28.0	.015333	.083122	.998	-.20853	.23920
dB	1.0	7.0	.205000*	.038297	.000	.10186	.30814
		28.0	.153000*	.038297	.002	.04986	.25614
		56.0	-.084333	.038297	.142	-.18748	.01881
	7.0	1.0	-.205000*	.038297	.000	-.30814	-.10186
		28.0	-.052000	.038297	.533	-.15514	.05114
		56.0	-.289333*	.038297	.000	-.39248	-.18619
	28.0	1.0	-.153000*	.038297	.002	-.25614	-.04986
		7.0	.052000	.038297	.533	-.05114	.15514
		56.0	-.237333*	.038297	.000	-.34048	-.13419
	56.0	1.0	.084333	.038297	.142	-.01881	.18748
		7.0	.289333*	.038297	.000	.18619	.39248
		28.0	.237333*	.038297	.000	.13419	.34048

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

TRD in distilled water

ANOVA

		Sum of Squares	df	Mean Square	F	Sig.
dE	Between Groups	64.258	3	21.419	240.274	.000
	Within Groups	3.209	36	.089		
	Total	67.468	39			
dL	Between Groups	81.095	3	27.032	269.823	.000
	Within Groups	3.607	36	.100		
	Total	84.702	39			
dA	Between Groups	3.695	3	1.232	24.932	.000
	Within Groups	1.779	36	.049		
	Total	5.474	39			
dB	Between Groups	12.755	3	4.252	83.484	.000
	Within Groups	1.833	36	.051		
	Total	14.589	39			

Multiple Comparisons

Tukey HSD

Dependent Variable	(I) Immertion_Time	(J) Immertion_Time	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval Lower Bound	95% Confidence Interval Upper Bound
dE	1.0	7.0	-1.054000*	.133526	.000	-1.41362	-.69438
		28.0	-3.043000*	.133526	.000	-3.40262	-2.68338
		56.0	-2.849000*	.133526	.000	-3.20862	-2.48938
	7.0	1.0	1.054000*	.133526	.000	.69438	1.41362
		28.0	-1.989000*	.133526	.000	-2.34862	-1.62938
		56.0	-1.795000*	.133526	.000	-2.15462	-1.43538
	28.0	1.0	3.043000*	.133526	.000	2.68338	3.40262
		7.0	1.989000*	.133526	.000	1.62938	2.34862
		56.0	.194000	.133526	.476	-.16562	.55362
	56.0	1.0	2.849000*	.133526	.000	2.48938	3.20862
		7.0	1.795000*	.133526	.000	1.43538	2.15462
		28.0	-.194000	.133526	.476	-.55362	.16562
dL	1.0	7.0	1.010000*	.141551	.000	.62877	1.39123
		28.0	3.250000*	.141551	.000	2.86877	3.63123
		56.0	3.273333*	.141551	.000	2.89210	3.65456
	7.0	1.0	-1.010000*	.141551	.000	-1.39123	-.62877
		28.0	2.240000*	.141551	.000	1.85877	2.62123
		56.0	2.263333*	.141551	.000	1.88210	2.64456
	28.0	1.0	-3.250000*	.141551	.000	-3.63123	-2.86877
		7.0	-2.240000*	.141551	.000	-2.62123	-1.85877
		56.0	.023333	.141551	.998	-.35790	.40456
	56.0	1.0	-3.273333*	.141551	.000	-3.65456	-2.89210
		7.0	-2.263333*	.141551	.000	-2.64456	-1.88210
		28.0	-.023333	.141551	.998	-.40456	.35790
dA	1.0	7.0	.521000*	.099402	.000	.25329	.78871
		28.0	.847000*	.099402	.000	.57929	1.11471
		56.0	.533333*	.099402	.000	.26562	.80105
	7.0	1.0	-.521000*	.099402	.000	-.78871	-.25329
		28.0	.326000*	.099402	.012	.05829	.59371
		56.0	.012333	.099402	.999	-.25538	.28005
	28.0	1.0	-.847000*	.099402	.000	-1.11471	-.57929
		7.0	-.326000*	.099402	.012	-.59371	-.05829
		56.0	-.313667*	.099402	.016	-.58138	-.04595
	56.0	1.0	-.533333*	.099402	.000	-.80105	-.26562
		7.0	-.012333	.099402	.999	-.28005	.25538
		28.0	.313667*	.099402	.016	.04595	.58138
dB	1.0	7.0	.970000*	.100924	.000	.69819	1.24181
		28.0	1.079000*	.100924	.000	.80719	1.35081
		56.0	-.188667	.100924	.259	-.46048	.08315
	7.0	1.0	-.970000*	.100924	.000	-1.24181	-.69819
		28.0	.109000	.100924	.704	-.16281	.38081
		56.0	-1.158667*	.100924	.000	-1.43048	-.88685
	28.0	1.0	-1.079000*	.100924	.000	-1.35081	-.80719
		7.0	-.109000	.100924	.704	-.38081	.16281
		56.0	-1.267667*	.100924	.000	-1.53948	-.99585
	56.0	1.0	.188667	.100924	.259	-.08315	.46048
		7.0	1.158667*	.100924	.000	.88685	1.43048
		28.0	1.267667*	.100924	.000	.99585	1.53948

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

TRX in dark chamber

ANOVA

		Sum of Squares	df	Mean Square	F	Sig.
dE	Between Groups	.317	3	.106	2.057	.123
	Within Groups	1.848	36	.051		
	Total	2.165	39			
dL	Between Groups	4.058	3	1.353	14.800	.000
	Within Groups	3.291	36	.091		
	Total	7.349	39			
dA	Between Groups	.080	3	.027	1.266	.301
	Within Groups	.760	36	.021		
	Total	.840	39			
dB	Between Groups	.012	3	.004	.228	.876
	Within Groups	.647	36	.018		
	Total	.659	39			

Multiple Comparisons

Tukey HSD

Dependent Variable	(I) Immertion_Time	(J) Immertion_Time	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
						Lower Bound	Upper Bound
dE	1.0	7.0	.249000	.101337	.084	-.02392	.52192
		28.0	.117000	.101337	.659	-.15592	.38992
		56.0	.092667	.101337	.797	-.18026	.36559
	7.0	1.0	-.249000	.101337	.084	-.52192	.02392
		28.0	-.132000	.101337	.567	-.40492	.14092
		56.0	-.156333	.101337	.423	-.42926	.11659
	28.0	1.0	-.117000	.101337	.659	-.38992	.15592
		7.0	.132000	.101337	.567	-.14092	.40492
		56.0	-.024333	.101337	.995	-.29726	.24859
	56.0	1.0	-.092667	.101337	.797	-.36559	.18026
		7.0	.156333	.101337	.423	-.11659	.42926
		28.0	.024333	.101337	.995	-.24859	.29726
dL	1.0	7.0	.431000*	.135209	.015	.06685	.79515
		28.0	.162000	.135209	.632	-.20215	.52615
		56.0	.841667*	.135209	.000	.47752	1.20581
	7.0	1.0	-.431000*	.135209	.015	-.79515	-.06685
		28.0	-.269000	.135209	.211	-.63315	.09515
		56.0	.410667*	.135209	.022	.04652	.77481
	28.0	1.0	-.162000	.135209	.632	-.52615	.20215
		7.0	.269000	.135209	.211	-.09515	.63315
		56.0	.679667*	.135209	.000	.31552	1.04381
	56.0	1.0	-.841667*	.135209	.000	-1.20581	-.47752
		7.0	-.410667*	.135209	.022	-.77481	-.04652
		28.0	-.679667*	.135209	.000	-1.04381	-.31552
dA	1.0	7.0	-.071000	.064958	.696	-.24595	.10395
		28.0	-.104333	.064958	.388	-.27928	.07061
		56.0	-.002667	.064958	1.000	-.17761	.17228
	7.0	1.0	.071000	.064958	.696	-.10395	.24595
		28.0	-.033333	.064958	.955	-.20828	.14161
		56.0	.068333	.064958	.720	-.10661	.24328
	28.0	1.0	.104333	.064958	.388	-.07061	.27928
		7.0	.033333	.064958	.955	-.14161	.20828
		56.0	.101667	.064958	.411	-.07328	.27661
	56.0	1.0	.002667	.064958	1.000	-.17228	.17761
		7.0	-.068333	.064958	.720	-.24328	.10661
		28.0	-.101667	.064958	.411	-.27661	.07328
dB	1.0	7.0	.047667	.059960	.856	-.11382	.20915
		28.0	.012667	.059960	.997	-.14882	.17415
		56.0	.024000	.059960	.978	-.13749	.18549
	7.0	1.0	-.047667	.059960	.856	-.20915	.11382
		28.0	-.035000	.059960	.936	-.19649	.12649
		56.0	-.023667	.059960	.979	-.18515	.13782
	28.0	1.0	-.012667	.059960	.997	-.17415	.14882
		7.0	.035000	.059960	.936	-.12649	.19649
		56.0	.011333	.059960	.998	-.15015	.17282
	56.0	1.0	-.024000	.059960	.978	-.18549	.13749
		7.0	.023667	.059960	.979	-.13782	.18515
		28.0	-.011333	.059960	.998	-.17282	.15015

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

BTC in dark chamber

ANOVA

		Sum of Squares	df	Mean Square	F	Sig.
dE	Between Groups	.348	3	.116	2.863	.050
	Within Groups	1.458	36	.040		
	Total	1.806	39			
dL	Between Groups	1.583	3	.528	2.863	.050
	Within Groups	6.637	36	.184		
	Total	8.220	39			
dA	Between Groups	.418	3	.139	2.982	.044
	Within Groups	1.684	36	.047		
	Total	2.102	39			
dB	Between Groups	.130	3	.043	2.715	.059
	Within Groups	.573	36	.016		
	Total	.702	39			

Multiple Comparisons

Tukey HSD

Dependent Variable	(I) Immertion_Time	(J) Immertion_Time	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
						Lower Bound	Upper Bound
dE	1.0	7.0	-.215667	.089999	.096	-.45806	.02672
		28.0	-.060000	.089999	.909	-.30239	.18239
		56.0	-.207400	.089999	.116	-.44979	.03499
	7.0	1.0	.215667	.089999	.096	-.02672	.45806
		28.0	.155667	.089999	.324	-.08672	.39806
		56.0	.008267	.089999	1.000	-.23412	.25066
	28.0	1.0	.060000	.089999	.909	-.18239	.30239
		7.0	-.155667	.089999	.324	-.39806	.08672
		56.0	-.147400	.089999	.371	-.38979	.09499
	56.0	1.0	.207400	.089999	.116	-.03499	.44979
		7.0	-.008267	.089999	1.000	-.25066	.23412
		28.0	.147400	.089999	.371	-.09499	.38979
dL	1.0	7.0	.167000	.192022	.820	-.35016	.68416
		28.0	.148667	.192022	.866	-.36849	.66583
		56.0	.539667*	.192022	.038	.02251	1.05683
	7.0	1.0	-.167000	.192022	.820	-.68416	.35016
		28.0	-.018333	.192022	1.000	-.53549	.49883
		56.0	.372667	.192022	.230	-.14449	.88983
	28.0	1.0	-.148667	.192022	.866	-.66583	.36849
		7.0	.018333	.192022	1.000	-.49883	.53549
		56.0	.391000	.192022	.194	-.12616	.90816
	56.0	1.0	-.539667*	.192022	.038	-1.05683	-.02251
		7.0	-.372667	.192022	.230	-.88983	.14449
		28.0	-.391000	.192022	.194	-.90816	.12616
dA	1.0	7.0	.184667	.096724	.242	-.07583	.44517
		28.0	.102667	.096724	.715	-.15783	.36317
		56.0	.277333*	.096724	.033	.01683	.53783
	7.0	1.0	-.184667	.096724	.242	-.44517	.07583
		28.0	-.082000	.096724	.831	-.34250	.17850
		56.0	.092667	.096724	.774	-.16783	.35317
	28.0	1.0	-.102667	.096724	.715	-.36317	.15783
		7.0	.082000	.096724	.831	-.17850	.34250
		56.0	.174667	.096724	.287	-.08583	.43517
	56.0	1.0	-.277333*	.096724	.033	-.53783	-.01683
		7.0	-.092667	.096724	.774	-.35317	.16783
		28.0	-.174667	.096724	.287	-.43517	.08583
dB	1.0	7.0	.138000	.056411	.086	-.01393	.28993
		28.0	.099667	.056411	.306	-.05226	.25159
		56.0	.018000	.056411	.989	-.13393	.16993
	7.0	1.0	-.138000	.056411	.086	-.28993	.01393
		28.0	-.038333	.056411	.904	-.19026	.11359
		56.0	-.120000	.056411	.164	-.27193	.03193
	28.0	1.0	-.099667	.056411	.306	-.25159	.05226
		7.0	.038333	.056411	.904	-.11359	.19026
		56.0	-.081667	.056411	.479	-.23359	.07026
	56.0	1.0	-.018000	.056411	.989	-.16993	.13393
		7.0	.120000	.056411	.164	-.03193	.27193
		28.0	.081667	.056411	.479	-.07026	.23359

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

TRD in dark chamber

ANOVA

		Sum of Squares	df	Mean Square	F	Sig.
dE	Between Groups	.055	3	.018	.552	.650
	Within Groups	1.193	36	.033		
	Total	1.247	39			
dL	Between Groups	1.397	3	.466	7.563	.000
	Within Groups	2.217	36	.062		
	Total	3.615	39			
dA	Between Groups	.349	3	.116	4.318	.011
	Within Groups	.970	36	.027		
	Total	1.319	39			
dB	Between Groups	.443	3	.148	6.378	.001
	Within Groups	.833	36	.023		
	Total	1.276	39			

Multiple Comparisons

Tukey HSD

Dependent Variable	(I) Immertion_Time	(J) Immertion_Time	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval Lower Bound	95% Confidence Interval Upper Bound
dE	1.0	7.0	-.074333	.081399	.798	-.29356	.14489
		28.0	.026667	.081399	.988	-.19256	.24589
		56.0	-.014000	.081399	.998	-.23323	.20523
	7.0	1.0	.074333	.081399	.798	-.14489	.29356
		28.0	.101000	.081399	.606	-.11823	.32023
		56.0	.060333	.081399	.880	-.15889	.27956
	28.0	1.0	-.026667	.081399	.988	-.24589	.19256
		7.0	-.101000	.081399	.606	-.32023	.11823
		56.0	-.040667	.081399	.959	-.25989	.17856
	56.0	1.0	.014000	.081399	.998	-.20523	.23323
		7.0	-.060333	.081399	.880	-.27956	.15889
		28.0	.040667	.081399	.959	-.17856	.25989
dL	1.0	7.0	.517333*	.110989	.000	.21841	.81625
		28.0	.344000*	.110989	.019	.04508	.64292
		56.0	.325000*	.110989	.029	.02608	.62392
	7.0	1.0	-.517333*	.110989	.000	-.81625	-.21841
		28.0	-.173333	.110989	.413	-.47225	.12559
		56.0	-.192333	.110989	.322	-.49125	.10659
	28.0	1.0	-.344000*	.110989	.019	-.64292	-.04508
		7.0	.173333	.110989	.413	-.12559	.47225
		56.0	-.019000	.110989	.998	-.31792	.27992
	56.0	1.0	-.325000*	.110989	.029	-.62392	-.02608
		7.0	.192333	.110989	.322	-.10659	.49125
		28.0	.019000	.110989	.998	-.27992	.31792
dA	1.0	7.0	.186000	.073407	.071	-.01170	.38370
		28.0	.171333	.073407	.109	-.02637	.36903
		56.0	.253333*	.073407	.008	.05563	.45103
	7.0	1.0	-.186000	.073407	.071	-.38370	.01170
		28.0	-.014667	.073407	.997	-.21237	.18303
		56.0	.067333	.073407	.796	-.13037	.26503
	28.0	1.0	-.171333	.073407	.109	-.36903	.02637
		7.0	.014667	.073407	.997	-.18303	.21237
		56.0	.082000	.073407	.682	-.11570	.27970
	56.0	1.0	-.253333*	.073407	.008	-.45103	-.05563
		7.0	-.067333	.073407	.796	-.26503	.13037
		28.0	-.082000	.073407	.682	-.27970	.11570
dB	1.0	7.0	.274667*	.068037	.001	.09143	.45791
		28.0	.123000	.068037	.286	-.06024	.30624
		56.0	.040000	.068037	.935	-.14324	.22324
	7.0	1.0	-.274667*	.068037	.001	-.45791	-.09143
		28.0	-.151667	.068037	.135	-.33491	.03157
		56.0	-.234667*	.068037	.008	-.41791	-.05143
	28.0	1.0	-.123000	.068037	.286	-.30624	.06024
		7.0	.151667	.068037	.135	-.03157	.33491
		56.0	-.083000	.068037	.619	-.26624	.10024
	56.0	1.0	-.040000	.068037	.935	-.22324	.14324
		7.0	.234667*	.068037	.008	.05143	.41791
		28.0	.083000	.068037	.619	-.10024	.26624

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

Each material in coffee solution at day 1

ANOVA

		Sum of Squares	df	Mean Square	F	Sig.
dE	Between Groups	.145	2	.072	1.364	.273
	Within Groups	1.432	27	.053		
	Total	1.577	29			
dL	Between Groups	.592	2	.296	4.925	.015
	Within Groups	1.624	27	.060		
	Total	2.216	29			
dA	Between Groups	.217	2	.108	2.325	.117
	Within Groups	1.257	27	.047		
	Total	1.474	29			
dB	Between Groups	.584	2	.292	7.051	.003
	Within Groups	1.118	27	.041		
	Total	1.702	29			

Multiple Comparisons

Tukey HSD

Dependent Variable	(I) Material_Types	(J) Material_Types	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
						Lower Bound	Upper Bound
dE	1.0	2.0	.079000	.103006	.726	-.17639	.33439
		3.0	-.091000	.103006	.655	-.34639	.16439
	2.0	1.0	-.079000	.103006	.726	-.33439	.17639
		3.0	-.170000	.103006	.243	-.42539	.08539
	3.0	1.0	.091000	.103006	.655	-.16439	.34639
		2.0	.170000	.103006	.243	-.08539	.42539
dL	1.0	2.0	-.294000*	.109666	.032	-.56591	-.02209
		3.0	-.302000*	.109666	.027	-.57391	-.03009
	2.0	1.0	.294000*	.109666	.032	.02209	.56591
		3.0	-.008000	.109666	.997	-.27991	.26391
	3.0	1.0	.302000*	.109666	.027	.03009	.57391
		2.0	.008000	.109666	.997	-.26391	.27991
dA	1.0	2.0	.085000	.096495	.657	-.15425	.32425
		3.0	.207000	.096495	.100	-.03225	.44625
	2.0	1.0	-.085000	.096495	.657	-.32425	.15425
		3.0	.122000	.096495	.427	-.11725	.36125
	3.0	1.0	-.207000	.096495	.100	-.44625	.03225
		2.0	-.122000	.096495	.427	-.36125	.11725
dB	1.0	2.0	-.081000	.090999	.651	-.30662	.14462
		3.0	-.328000*	.090999	.003	-.55362	-.10238
	2.0	1.0	.081000	.090999	.651	-.14462	.30662
		3.0	-.247000*	.090999	.030	-.47262	-.02138
	3.0	1.0	.328000*	.090999	.003	.10238	.55362
		2.0	.247000*	.090999	.030	.02138	.47262

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

Each material in distilled water at day 1

ANOVA

		Sum of Squares	df	Mean Square	F	Sig.
dE	Between Groups	.170	2	.085	1.814	.182
	Within Groups	1.263	27	.047		
	Total	1.433	29			
dL	Between Groups	.410	2	.205	1.692	.203
	Within Groups	3.267	27	.121		
	Total	3.677	29			
dA	Between Groups	.680	2	.340	14.946	.000
	Within Groups	.614	27	.023		
	Total	1.294	29			
dB	Between Groups	.103	2	.052	2.044	.149
	Within Groups	.683	27	.025		
	Total	.787	29			

Multiple Comparisons

Tukey HSD

Dependent Variable	(I) Material_Types	(J) Material_Types	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval Lower Bound	Upper Bound
dE	1.0	2.0	.183000	.096732	.160	-.05684	.42284
		3.0	.110000	.096732	.500	-.12984	.34984
	2.0	1.0	-.183000	.096732	.160	-.42284	.05684
		3.0	-.073000	.096732	.733	-.31284	.16684
	3.0	1.0	-.110000	.096732	.500	-.34984	.12984
		2.0	.073000	.096732	.733	-.16684	.31284
dL	1.0	2.0	-.015000	.155571	.995	-.40072	.37072
		3.0	-.255000	.155571	.247	-.64072	.13072
	2.0	1.0	.015000	.155571	.995	-.37072	.40072
		3.0	-.240000	.155571	.288	-.62572	.14572
	3.0	1.0	.255000	.155571	.247	-.13072	.64072
		2.0	.240000	.155571	.288	-.14572	.62572
dA	1.0	2.0	-.238000*	.067459	.004	-.40526	-.07074
		3.0	-.363000*	.067459	.000	-.53026	-.19574
	2.0	1.0	.238000*	.067459	.004	.07074	.40526
		3.0	-.125000	.067459	.172	-.29226	.04226
	3.0	1.0	.363000*	.067459	.000	.19574	.53026
		2.0	.125000	.067459	.172	-.04226	.29226
dB	1.0	2.0	-.131000	.071152	.176	-.30742	.04542
		3.0	-.014000	.071152	.979	-.19042	.16242
	2.0	1.0	.131000	.071152	.176	-.04542	.30742
		3.0	.117000	.071152	.245	-.05942	.29342
	3.0	1.0	.014000	.071152	.979	-.16242	.19042
		2.0	-.117000	.071152	.245	-.29342	.05942

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

Each material in dark chamber at day 1

ANOVA

		Sum of Squares	df	Mean Square	F	Sig.
dE	Between Groups	.187	2	.093	2.508	.100
	Within Groups	1.006	27	.037		
	Total	1.193	29			
dL	Between Groups	1.721	2	.860	9.652	.001
	Within Groups	2.407	27	.089		
	Total	4.127	29			
dA	Between Groups	.876	2	.438	20.799	.000
	Within Groups	.569	27	.021		
	Total	1.444	29			
dB	Between Groups	.114	2	.057	4.465	.021
	Within Groups	.346	27	.013		
	Total	.461	29			

Multiple Comparisons

Tukey HSD

Dependent Variable	(I) Material_Types	(J) Material_Types	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
						Lower Bound	Upper Bound
dE	1.0	2.0	.187000	.086326	.096	-.02704	.40104
		3.0	.136000	.086326	.273	-.07804	.35004
	2.0	1.0	-.187000	.086326	.096	-.40104	.02704
		3.0	-.051000	.086326	.826	-.26504	.16304
	3.0	1.0	-.136000	.086326	.273	-.35004	.07804
		2.0	.051000	.086326	.826	-.16304	.26504
dL	1.0	2.0	.584667*	.133519	.000	.25362	.91572
		3.0	.334000*	.133519	.048	.00295	.66505
	2.0	1.0	-.584667*	.133519	.000	-.91572	-.25362
		3.0	-.250667	.133519	.165	-.58172	.08038
	3.0	1.0	-.334000*	.133519	.048	-.66505	-.00295
		2.0	.250667	.133519	.165	-.08038	.58172
dA	1.0	2.0	-.177333*	.064895	.029	-.33823	-.01643
		3.0	-.417000*	.064895	.000	-.57790	-.25610
	2.0	1.0	.177333*	.064895	.029	.01643	.33823
		3.0	-.239667*	.064895	.003	-.40057	-.07877
	3.0	1.0	.417000*	.064895	.000	.25610	.57790
		2.0	.239667*	.064895	.003	.07877	.40057
dB	1.0	2.0	-.123667	.050634	.054	-.24921	.00188
		3.0	-.137333*	.050634	.030	-.26288	-.01179
	2.0	1.0	.123667	.050634	.054	-.00188	.24921
		3.0	-.013667	.050634	.961	-.13921	.11188
	3.0	1.0	.137333*	.050634	.030	.01179	.26288
		2.0	.013667	.050634	.961	-.11188	.13921

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

Each material in coffee solution at day 7

ANOVA

		Sum of Squares	df	Mean Square	F	Sig.
dE	Between Groups	43.901	2	21.950	115.885	.000
	Within Groups	5.114	27	.189		
	Total	49.015	29			
dL	Between Groups	11.905	2	5.952	37.242	.000
	Within Groups	4.316	27	.160		
	Total	16.220	29			
dA	Between Groups	6.846	2	3.423	36.788	.000
	Within Groups	2.512	27	.093		
	Total	9.359	29			
dB	Between Groups	31.313	2	15.656	48.963	.000
	Within Groups	8.633	27	.320		
	Total	39.946	29			

Multiple Comparisons

Tukey HSD

Dependent Variable	(I) Material_Types	(J) Material_Types	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval Lower Bound	95% Confidence Interval Upper Bound
dE	1.0	2.0	-2.550000*	.194636	.000	-3.03258	-2.06742
		3.0	-2.582000*	.194636	.000	-3.06458	-2.09942
	2.0	1.0	2.550000*	.194636	.000	2.06742	3.03258
		3.0	-.032000	.194636	.985	-.51458	.45058
	3.0	1.0	2.582000*	.194636	.000	2.09942	3.06458
		2.0	.032000	.194636	.985	-.45058	.51458
dL	1.0	2.0	1.003000*	.178792	.000	.55970	1.44630
		3.0	1.517000*	.178792	.000	1.07370	1.96030
	2.0	1.0	-1.003000*	.178792	.000	-1.44630	-.55970
		3.0	.514000*	.178792	.021	.07070	.95730
	3.0	1.0	-1.517000*	.178792	.000	-1.96030	-1.07370
		2.0	-.514000*	.178792	.021	-.95730	-.07070
dA	1.0	2.0	.997000*	.136418	.000	.65876	1.33524
		3.0	1.029000*	.136418	.000	.69076	1.36724
	2.0	1.0	-.997000*	.136418	.000	-1.33524	-.65876
		3.0	.032000	.136418	.970	-.30624	.37024
	3.0	1.0	-1.029000*	.136418	.000	-1.36724	-.69076
		2.0	-.032000	.136418	.970	-.37024	.30624
dB	1.0	2.0	-2.316000*	.252886	.000	-2.94301	-1.68899
		3.0	-1.979000*	.252886	.000	-2.60601	-1.35199
	2.0	1.0	2.316000*	.252886	.000	1.68899	2.94301
		3.0	.337000	.252886	.390	-.29001	.96401
	3.0	1.0	1.979000*	.252886	.000	1.35199	2.60601
		2.0	-.337000	.252886	.390	-.96401	.29001

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

Each material in distilled water at day 7

ANOVA

		Sum of Squares	df	Mean Square	F	Sig.
dE	Between Groups	6.191	2	3.096	49.175	.000
	Within Groups	1.637	26	.063		
	Total	7.828	28			
dL	Between Groups	4.797	2	2.399	18.876	.000
	Within Groups	3.431	27	.127		
	Total	8.228	29			
dA	Between Groups	.595	2	.298	8.874	.001
	Within Groups	.906	27	.034		
	Total	1.501	29			
dB	Between Groups	6.561	2	3.280	212.852	.000
	Within Groups	.416	27	.015		
	Total	6.977	29			

Multiple Comparisons

Tukey HSD

Dependent Variable	(I) Material_Types	(J) Material_Types	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
						Lower Bound	Upper Bound
dE	1.0	2.0	-.219556	.115280	.158	-.50601	.06690
		3.0	-1.058000*	.112205	.000	-1.33682	-.77918
	2.0	1.0	.219556	.115280	.158	-.06690	.50601
		3.0	-.838444*	.115280	.000	-1.12490	-.55199
	3.0	1.0	1.058000*	.112205	.000	.77918	1.33682
		2.0	.838444*	.115280	.000	.55199	1.12490
dL	1.0	2.0	.693000*	.159418	.001	.29774	1.08826
		3.0	.946000*	.159418	.000	.55074	1.34126
	2.0	1.0	-.693000*	.159418	.001	-1.08826	-.29774
		3.0	.253000	.159418	.268	-.14226	.64826
	3.0	1.0	-.946000*	.159418	.000	-1.34126	-.55074
		2.0	-.253000	.159418	.268	-.64826	.14226
dA	1.0	2.0	.307000*	.081915	.002	.10390	.51010
		3.0	.290000*	.081915	.004	.08690	.49310
	2.0	1.0	-.307000*	.081915	.002	-.51010	-.10390
		3.0	-.017000	.081915	.977	-.22010	.18610
	3.0	1.0	-.290000*	.081915	.004	-.49310	-.08690
		2.0	.017000	.081915	.977	-.18610	.22010
dB	1.0	2.0	.192000*	.055519	.005	.05434	.32966
		3.0	1.074000*	.055519	.000	.93634	1.21166
	2.0	1.0	-.192000*	.055519	.005	-.32966	-.05434
		3.0	.882000*	.055519	.000	.74434	1.01966
	3.0	1.0	-1.074000*	.055519	.000	-1.21166	-.93634
		2.0	-.882000*	.055519	.000	-1.01966	-.74434

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

Each material in dark chamber at day 7

ANOVA

		Sum of Squares	df	Mean Square	F	Sig.
dE	Between Groups	.401	2	.201	4.444	.021
	Within Groups	1.219	27	.045		
	Total	1.620	29			
dL	Between Groups	.965	2	.482	4.430	.022
	Within Groups	2.940	27	.109		
	Total	3.905	29			
dA	Between Groups	.295	2	.148	3.514	.044
	Within Groups	1.134	27	.042		
	Total	1.429	29			
dB	Between Groups	.081	2	.040	3.559	.042
	Within Groups	.307	27	.011		
	Total	.388	29			

Multiple Comparisons

Tukey HSD

Dependent Variable	(I) Material_Types	(J) Material_Types	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval Lower Bound	95% Confidence Interval Upper Bound
dE	1.0	2.0	-.277667*	.095008	.018	-.51323	-.04210
		3.0	-.187333	.095008	.139	-.42290	.04823
	2.0	1.0	.277667*	.095008	.018	.04210	.51323
		3.0	.090333	.095008	.614	-.14523	.32590
	3.0	1.0	.187333	.095008	.139	-.04823	.42290
		2.0	-.090333	.095008	.614	-.32590	.14523
dL	1.0	2.0	.320667	.147577	.094	-.04524	.68657
		3.0	.420333*	.147577	.022	.05443	.78624
	2.0	1.0	-.320667	.147577	.094	-.68657	.04524
		3.0	.099667	.147577	.780	-.26624	.46557
	3.0	1.0	-.420333*	.147577	.022	-.78624	-.05443
		2.0	-.099667	.147577	.780	-.46557	.26624
dA	1.0	2.0	.078333	.091646	.673	-.14889	.30556
		3.0	-.160000	.091646	.207	-.38723	.06723
	2.0	1.0	-.078333	.091646	.673	-.30556	.14889
		3.0	-.238333*	.091646	.038	-.46556	-.01111
	3.0	1.0	.160000	.091646	.207	-.06723	.38723
		2.0	.238333*	.091646	.038	.01111	.46556
dB	1.0	2.0	-.033333	.047687	.766	-.15157	.08490
		3.0	.089667	.047687	.164	-.02857	.20790
	2.0	1.0	.033333	.047687	.766	-.08490	.15157
		3.0	.123000*	.047687	.040	.00476	.24124
	3.0	1.0	-.089667	.047687	.164	-.20790	.02857
		2.0	-.123000*	.047687	.040	-.24124	-.00476

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

Each material in coffee solution at day 28

ANOVA

		Sum of Squares	df	Mean Square	F	Sig.
dE	Between Groups	427.163	2	213.582	90.497	.000
	Within Groups	63.723	27	2.360		
	Total	490.886	29			
dL	Between Groups	254.187	2	127.094	90.299	.000
	Within Groups	38.002	27	1.407		
	Total	292.189	29			
dA	Between Groups	42.259	2	21.130	53.889	.000
	Within Groups	10.587	27	.392		
	Total	52.846	29			
dB	Between Groups	147.626	2	73.813	61.601	.000
	Within Groups	32.352	27	1.198		
	Total	179.978	29			

Multiple Comparisons

Tukey HSD

Dependent Variable	(I) Material_Types	(J) Material_Types	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval Lower Bound	95% Confidence Interval Upper Bound
dE	1.0	2.0	-6.012000*	.687038	.000	-7.71545	-4.30855
		3.0	-9.086000*	.687038	.000	-10.78945	-7.38255
	2.0	1.0	6.012000*	.687038	.000	4.30855	7.71545
		3.0	-3.074000*	.687038	.000	-4.77745	-1.37055
	3.0	1.0	9.086000*	.687038	.000	7.38255	10.78945
		2.0	3.074000*	.687038	.000	1.37055	4.77745
dL	1.0	2.0	3.670000*	.530562	.000	2.35451	4.98549
		3.0	7.129000*	.530562	.000	5.81351	8.44449
	2.0	1.0	-3.670000*	.530562	.000	-4.98549	-2.35451
		3.0	3.459000*	.530562	.000	2.14351	4.77449
	3.0	1.0	-7.129000*	.530562	.000	-8.44449	-5.81351
		2.0	-3.459000*	.530562	.000	-4.77449	-2.14351
dA	1.0	2.0	1.615000*	.280034	.000	.92068	2.30932
		3.0	2.901000*	.280034	.000	2.20668	3.59532
	2.0	1.0	-1.615000*	.280034	.000	-2.30932	-.92068
		3.0	1.286000*	.280034	.000	.59168	1.98032
	3.0	1.0	-2.901000*	.280034	.000	-3.59532	-2.20668
		2.0	-1.286000*	.280034	.000	-1.98032	-.59168
dB	1.0	2.0	-4.548000*	.489538	.000	-5.76177	-3.33423
		3.0	-4.849000*	.489538	.000	-6.06277	-3.63523
	2.0	1.0	4.548000*	.489538	.000	3.33423	5.76177
		3.0	-.301000	.489538	.813	-1.51477	.91277
	3.0	1.0	4.849000*	.489538	.000	3.63523	6.06277
		2.0	.301000	.489538	.813	-.91277	1.51477

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

Each material in distilled water at day 28

ANOVA

		Sum of Squares	df	Mean Square	F	Sig.
dE	Between Groups	48.937	2	24.469	290.627	.000
	Within Groups	2.273	27	.084		
	Total	51.210	29			
dL	Between Groups	72.014	2	36.007	301.096	.000
	Within Groups	3.229	27	.120		
	Total	75.243	29			
dA	Between Groups	3.371	2	1.685	46.257	.000
	Within Groups	.984	27	.036		
	Total	4.354	29			
dB	Between Groups	11.076	2	5.538	160.012	.000
	Within Groups	.934	27	.035		
	Total	12.010	29			

Multiple Comparisons

Tukey HSD

Dependent Variable	(I) Material_Types	(J) Material_Types	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
						Lower Bound	Upper Bound
dE	1.0	2.0	.050000	.129763	.922	-.27174	.37174
		3.0	-2.684000*	.129763	.000	-3.00574	-2.36226
	2.0	1.0	-.050000	.129763	.922	-.37174	.27174
		3.0	-2.734000*	.129763	.000	-3.05574	-2.41226
	3.0	1.0	2.684000*	.129763	.000	2.36226	3.00574
		2.0	2.734000*	.129763	.000	2.41226	3.05574
dL	1.0	2.0	1.106000*	.154653	.000	.72255	1.48945
		3.0	3.697000*	.154653	.000	3.31355	4.08045
	2.0	1.0	-1.106000*	.154653	.000	-1.48945	-.72255
		3.0	2.591000*	.154653	.000	2.20755	2.97445
	3.0	1.0	-3.697000*	.154653	.000	-4.08045	-3.31355
		2.0	-2.591000*	.154653	.000	-2.97445	-2.20755
dA	1.0	2.0	.500000*	.085362	.000	.28835	.71165
		3.0	.814000*	.085362	.000	.60235	1.02565
	2.0	1.0	-.500000*	.085362	.000	-.71165	-.28835
		3.0	.314000*	.085362	.003	.10235	.52565
	3.0	1.0	-.814000*	.085362	.000	-1.02565	-.60235
		2.0	-.314000*	.085362	.003	-.52565	-.10235
dB	1.0	2.0	.398000*	.083198	.000	.19172	.60428
		3.0	1.441000*	.083198	.000	1.23472	1.64728
	2.0	1.0	-.398000*	.083198	.000	-.60428	-.19172
		3.0	1.043000*	.083198	.000	.83672	1.24928
	3.0	1.0	-1.441000*	.083198	.000	-1.64728	-1.23472
		2.0	-1.043000*	.083198	.000	-1.24928	-.83672

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

Each material in dark chamber at day 28

ANOVA

		Sum of Squares	df	Mean Square	F	Sig.
dE	Between Groups	.012	2	.006	.116	.891
	Within Groups	1.339	27	.050		
	Total	1.350	29			
dL	Between Groups	1.986	2	.993	9.358	.001
	Within Groups	2.865	27	.106		
	Total	4.851	29			
dA	Between Groups	.167	2	.083	3.556	.043
	Within Groups	.634	27	.023		
	Total	.801	29			
dB	Between Groups	.007	2	.004	.200	.820
	Within Groups	.488	27	.018		
	Total	.495	29			

Multiple Comparisons

Tukey HSD

Dependent Variable	(I) Material_Types	(J) Material_Types	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
						Lower Bound	Upper Bound
dE	1.0	2.0	.010000	.099575	.994	-.23689	.25689
		3.0	.045667	.099575	.891	-.20122	.29256
	2.0	1.0	-.010000	.099575	.994	-.25689	.23689
		3.0	.035667	.099575	.932	-.21122	.28256
	3.0	1.0	-.045667	.099575	.891	-.29256	.20122
		2.0	-.035667	.099575	.932	-.28256	.21122
dL	1.0	2.0	.571333*	.145673	.002	.21015	.93252
		3.0	.516000*	.145673	.004	.15482	.87718
	2.0	1.0	-.571333*	.145673	.002	-.93252	-.21015
		3.0	-.055333	.145673	.924	-.41652	.30585
	3.0	1.0	-.516000*	.145673	.004	-.87718	-.15482
		2.0	.055333	.145673	.924	-.30585	.41652
dA	1.0	2.0	.029667	.068528	.902	-.14024	.19958
		3.0	-.141333	.068528	.117	-.31124	.02858
	2.0	1.0	-.029667	.068528	.902	-.19958	.14024
		3.0	-.171000*	.068528	.048	-.34091	-.00109
	3.0	1.0	.141333	.068528	.117	-.02858	.31124
		2.0	.171000*	.068528	.048	.00109	.34091
dB	1.0	2.0	-.036667	.060131	.816	-.18576	.11242
		3.0	-.027000	.060131	.895	-.17609	.12209
	2.0	1.0	.036667	.060131	.816	-.11242	.18576
		3.0	.009667	.060131	.986	-.13942	.15876
	3.0	1.0	.027000	.060131	.895	-.12209	.17609
		2.0	-.009667	.060131	.986	-.15876	.13942

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

Each material in coffee solution at day 56

ANOVA

		Sum of Squares	df	Mean Square	F	Sig.
dE	Between Groups	1346.382	2	673.191	91.732	.000
	Within Groups	198.143	27	7.339		
	Total	1544.526	29			
dL	Between Groups	933.249	2	466.625	84.557	.000
	Within Groups	148.998	27	5.518		
	Total	1082.247	29			
dA	Between Groups	73.852	2	36.926	32.342	.000
	Within Groups	30.827	27	1.142		
	Total	104.678	29			
dB	Between Groups	460.029	2	230.014	75.688	.000
	Within Groups	82.052	27	3.039		
	Total	542.081	29			

Multiple Comparisons

Tukey HSD

Dependent Variable	(I) Material_Types	(J) Material_Types	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval Lower Bound	95% Confidence Interval Upper Bound
dE	1.0	2.0	-8.117000*	1.211499	.000	-11.12081	-5.11319
		3.0	-16.409333*	1.211499	.000	-19.41314	-13.40552
	2.0	1.0	8.117000*	1.211499	.000	5.11319	11.12081
		3.0	-8.292333*	1.211499	.000	-11.29614	-5.28852
	3.0	1.0	16.409333*	1.211499	.000	13.40552	19.41314
		2.0	8.292333*	1.211499	.000	5.28852	11.29614
dL	1.0	2.0	5.322000*	1.050566	.000	2.71721	7.92679
		3.0	13.558000*	1.050566	.000	10.95321	16.16279
	2.0	1.0	-5.322000*	1.050566	.000	-7.92679	-2.71721
		3.0	8.236000*	1.050566	.000	5.63121	10.84079
	3.0	1.0	-13.558000*	1.050566	.000	-16.16279	-10.95321
		2.0	-8.236000*	1.050566	.000	-10.84079	-5.63121
dA	1.0	2.0	1.957333*	.477855	.001	.77253	3.14214
		3.0	3.843000*	.477855	.000	2.65820	5.02780
	2.0	1.0	-1.957333*	.477855	.001	-3.14214	-.77253
		3.0	1.885667*	.477855	.001	.70086	3.07047
	3.0	1.0	-3.843000*	.477855	.000	-5.02780	-2.65820
		2.0	-1.885667*	.477855	.001	-3.07047	-.70086
dB	1.0	2.0	-6.967333*	.779612	.000	-8.90032	-5.03435
		3.0	-9.193000*	.779612	.000	-11.12598	-7.26002
	2.0	1.0	6.967333*	.779612	.000	5.03435	8.90032
		3.0	-2.225667*	.779612	.022	-4.15865	-.29268
	3.0	1.0	9.193000*	.779612	.000	7.26002	11.12598
		2.0	2.225667*	.779612	.022	.29268	4.15865

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

Each material in distilled water at day 56

ANOVA

		Sum of Squares	df	Mean Square	F	Sig.
dE	Between Groups	42.742	2	21.371	105.575	.000
	Within Groups	5.465	27	.202		
	Total	48.207	29			
dL	Between Groups	45.202	2	22.601	96.018	.000
	Within Groups	6.355	27	.235		
	Total	51.557	29			
dA	Between Groups	.210	2	.105	1.426	.258
	Within Groups	1.985	27	.074		
	Total	2.195	29			
dB	Between Groups	.033	2	.016	.257	.775
	Within Groups	1.724	27	.064		
	Total	1.757	29			

Multiple Comparisons

Tukey HSD

Dependent Variable	(I) Material_Types	(J) Material_Types	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
						Lower Bound	Upper Bound
dE	1.0	2.0	.018000	.201208	.996	-.48088	.51688
		3.0	-2.523000*	.201208	.000	-3.02188	-2.02412
	2.0	1.0	-.018000	.201208	.996	-.51688	.48088
		3.0	-2.541000*	.201208	.000	-3.03988	-2.04212
	3.0	1.0	2.523000*	.201208	.000	2.02412	3.02188
		2.0	2.541000*	.201208	.000	2.04212	3.03988
dL	1.0	2.0	-.040667	.216972	.981	-.57863	.49730
		3.0	2.583333*	.216972	.000	2.04537	3.12130
	2.0	1.0	.040667	.216972	.981	-.49730	.57863
		3.0	2.624000*	.216972	.000	2.08604	3.16196
	3.0	1.0	-2.583333*	.216972	.000	-3.12130	-2.04537
		2.0	-2.624000*	.216972	.000	-3.16196	-2.08604
dA	1.0	2.0	.169000	.121273	.358	-.13169	.46969
		3.0	.184667	.121273	.296	-.11602	.48535
	2.0	1.0	-.169000	.121273	.358	-.46969	.13169
		3.0	.015667	.121273	.991	-.28502	.31635
	3.0	1.0	-.184667	.121273	.296	-.48535	.11602
		2.0	-.015667	.121273	.991	-.31635	.28502
dB	1.0	2.0	-.075667	.113005	.783	-.35585	.20452
		3.0	-.063000	.113005	.844	-.34319	.21719
	2.0	1.0	.075667	.113005	.783	-.20452	.35585
		3.0	.012667	.113005	.993	-.26752	.29285
	3.0	1.0	.063000	.113005	.844	-.21719	.34319
		2.0	-.012667	.113005	.993	-.29285	.26752

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

Each material in dark chamber at day 56

ANOVA

		Sum of Squares	df	Mean Square	F	Sig.
dE	Between Groups	.012	2	.006	.191	.827
	Within Groups	.861	27	.032		
	Total	.873	29			
dL	Between Groups	1.099	2	.550	3.773	.036
	Within Groups	3.933	27	.146		
	Total	5.033	29			
dA	Between Groups	.353	2	.177	4.428	.022
	Within Groups	1.077	27	.040		
	Total	1.430	29			
dB	Between Groups	.105	2	.053	1.560	.229
	Within Groups	.912	27	.034		
	Total	1.017	29			

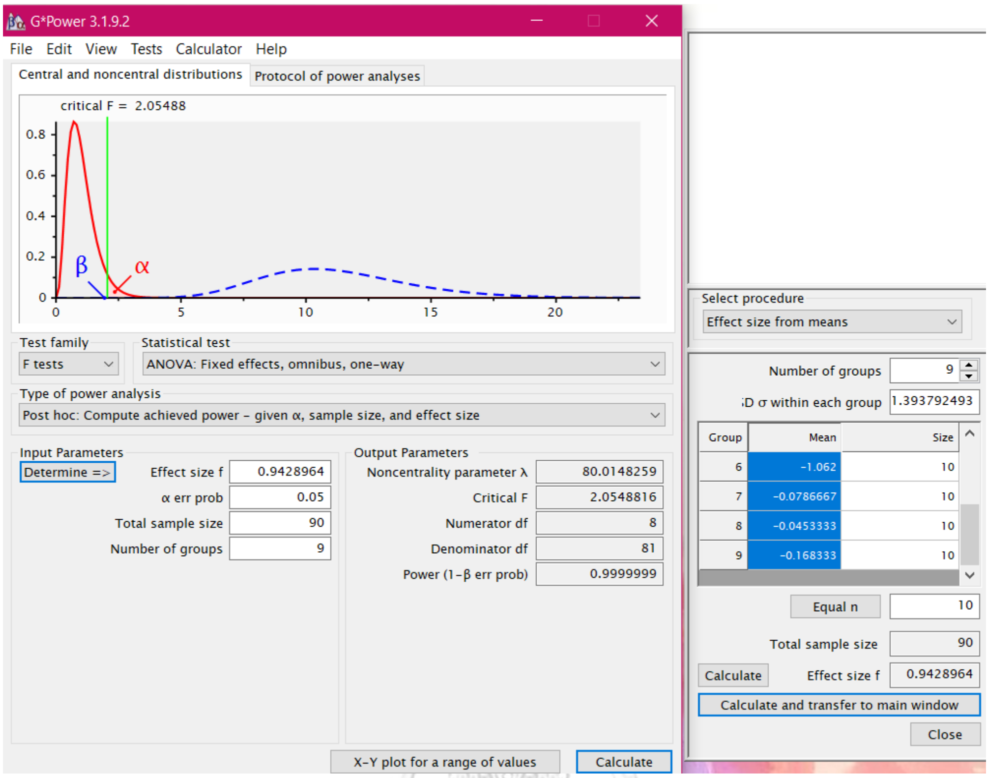
Multiple Comparisons

Tukey HSD

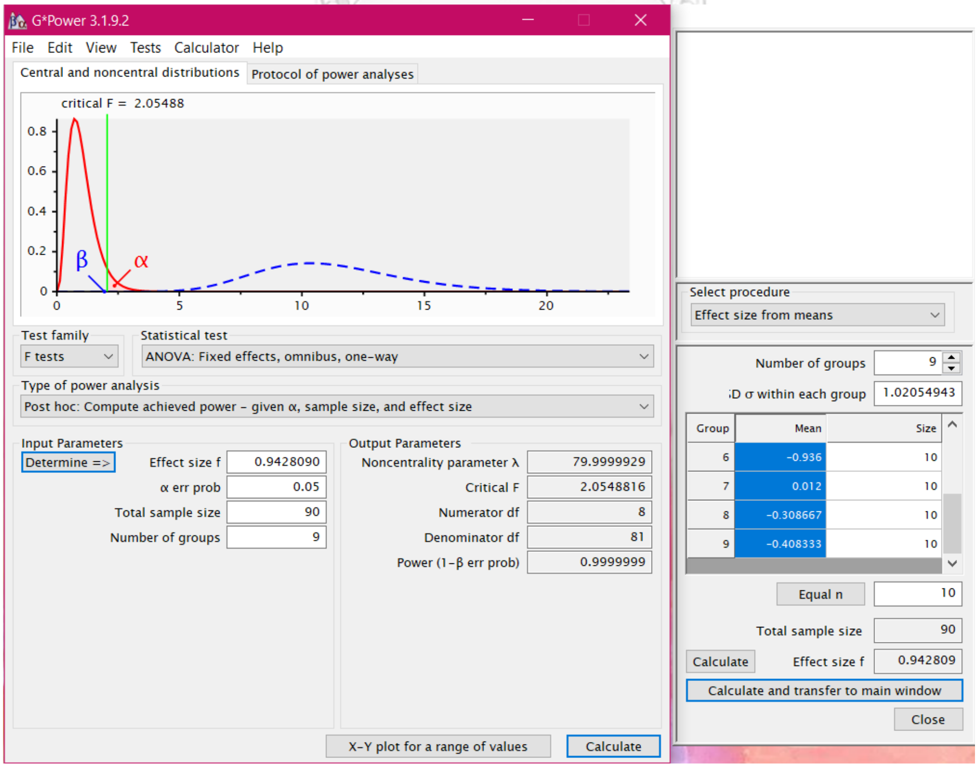
Dependent Variable	(I) Material_Types	(J) Material_Types	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
						Lower Bound	Upper Bound
dE	1.0	2.0	-.019667	.079849	.967	-.21765	.17831
		3.0	.029333	.079849	.929	-.16865	.22731
	2.0	1.0	.019667	.079849	.967	-.17831	.21765
		3.0	.049000	.079849	.814	-.14898	.24698
	3.0	1.0	-.029333	.079849	.929	-.22731	.16865
		2.0	-.049000	.079849	.814	-.24698	.14898
dL	1.0	2.0	.282667	.170694	.240	-.14055	.70589
		3.0	-.182667	.170694	.540	-.60589	.24055
	2.0	1.0	-.282667	.170694	.240	-.70589	.14055
		3.0	-.465333*	.170694	.029	-.88855	-.04211
	3.0	1.0	.182667	.170694	.540	-.24055	.60589
		2.0	.465333*	.170694	.029	.04211	.88855
dA	1.0	2.0	.102667	.089322	.493	-.11880	.32413
		3.0	-.161000	.089322	.188	-.38247	.06047
	2.0	1.0	-.102667	.089322	.493	-.32413	.11880
		3.0	-.263667*	.089322	.017	-.48513	-.04220
	3.0	1.0	.161000	.089322	.188	-.06047	.38247
		2.0	.263667*	.089322	.017	.04220	.48513
dB	1.0	2.0	-.129667	.082188	.272	-.33345	.07411
		3.0	-.121333	.082188	.318	-.32511	.08245
	2.0	1.0	.129667	.082188	.272	-.07411	.33345
		3.0	.008333	.082188	.994	-.19545	.21211
	3.0	1.0	.121333	.082188	.318	-.08245	.32511
		2.0	-.008333	.082188	.994	-.21211	.19545

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

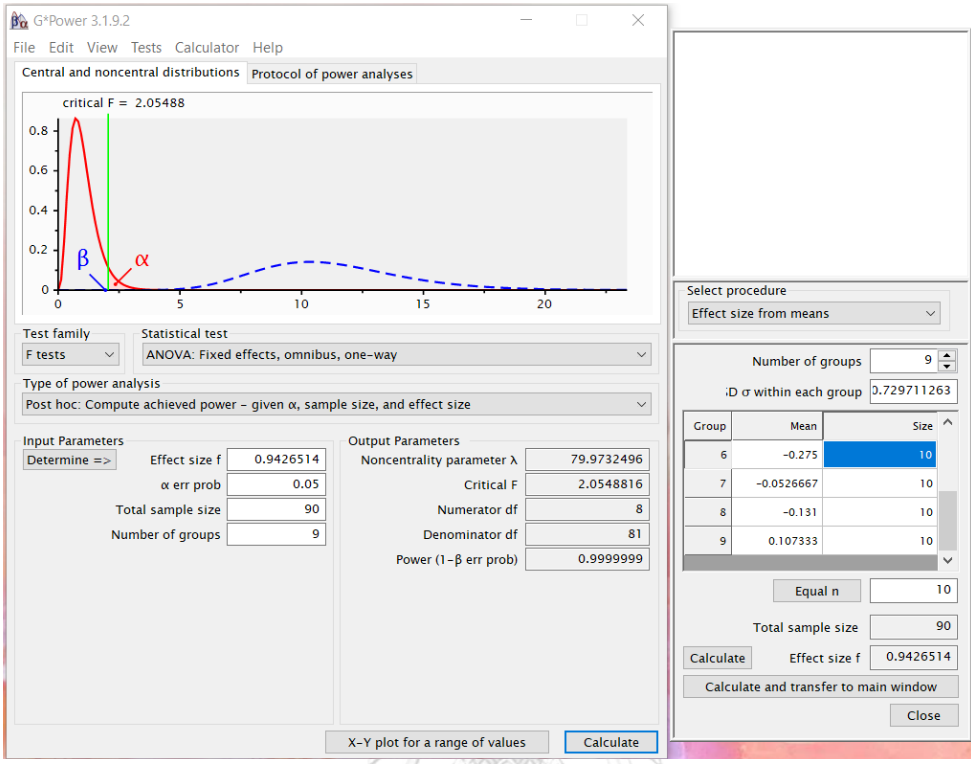
Power analysis of ΔE* outcome variable (Power = 0.99)



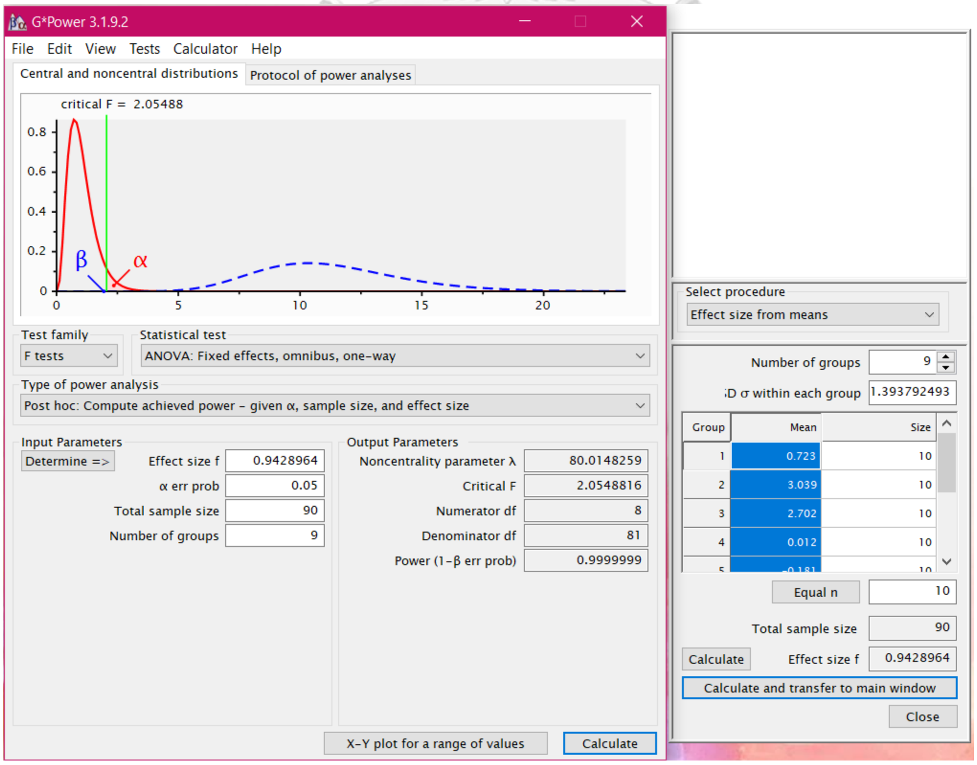
Power analysis of ΔL* outcome variable (Power = 0.99)



Power analysis of Δa* outcome variable (Power = 0.99)



Power analysis of Δb* outcome variable (Power = 0.99)



Repeated mixed Analysis of variance: Nanohardness

Within-Subjects Factors

Measure: Hardness

time	Dependent Variable
1	H_day1
2	H_day56

Between-Subjects Factors

		N
Material	1.0	27
	2.0	27
	3.0	27
Solution	1.0	27
	2.0	27
	3.0	27

Box's Test of Equality of Covariance Matrices[a]

Box's M	134.680
F	5.040
df1	24
df2	15845.434
Sig.	.000

Tests the null hypothesis that the observed covariance matrices of the dependent variables are equal across groups.

a. Design: Intercept + Material + Solution + Material * Solution
Within Subjects Design: time

Descriptive Statistics

	Material	Solution	Mean	Std. Deviation	N
H_day1	1.0	1.0	.17188889	.003218868	9
		2.0	.15044444	.023163069	9
		3.0	.16966667	.007615773	9
		Total	.16400000	.016804303	27
	2.0	1.0	.25368356	.009525759	9
		2.0	.24386422	.007713615	9
		3.0	.24010944	.008808334	9
		Total	.24588574	.010203574	27
	3.0	1.0	.22362800	.022699990	9
		2.0	.24404300	.024761069	9
		3.0	.24885800	.036557034	9
		Total	.23884300	.029708884	27
	Total	1.0	.21640015	.037077385	27
		2.0	.21278389	.048886460	27
		3.0	.21954470	.041927592	27
		Total	.21624291	.042456469	81
H_day56	1.0	1.0	.06466500	.010813245	9
		2.0	.10985556	.042858236	9
		3.0	.17650122	.015525511	9
		Total	.11700726	.053540741	27
	2.0	1.0	.09865844	.033684914	9
		2.0	.19338011	.022726140	9
		3.0	.23804622	.012563431	9
		Total	.17669493	.063748469	27
	3.0	1.0	.08381878	.048656081	9
		2.0	.15969889	.026511724	9
		3.0	.20973133	.025862586	9
		Total	.15108300	.062713726	27
	Total	1.0	.08238074	.036257638	27
		2.0	.15431152	.046506749	27
		3.0	.20809293	.031392869	27
		Total	.14826173	.064310617	81

Multivariate Tests[a]

Effect		Value	F	Hypothesis df	Error df	Sig.	Partial Eta Squared
time	Pillai's Trace	.832	356.218[b]	1.000	72.000	.000	.832
	Wilks' Lambda	.168	356.218[b]	1.000	72.000	.000	.832
	Hotelling's Trace	4.947	356.218[b]	1.000	72.000	.000	.832
	Roy's Largest Root	4.947	356.218[b]	1.000	72.000	.000	.832
time * Material	Pillai's Trace	.229	10.703[b]	2.000	72.000	.000	.229
	Wilks' Lambda	.771	10.703[b]	2.000	72.000	.000	.229
	Hotelling's Trace	.297	10.703[b]	2.000	72.000	.000	.229
	Roy's Largest Root	.297	10.703[b]	2.000	72.000	.000	.229
time * Solution	Pillai's Trace	.732	98.238[b]	2.000	72.000	.000	.732
	Wilks' Lambda	.268	98.238[b]	2.000	72.000	.000	.732
	Hotelling's Trace	2.729	98.238[b]	2.000	72.000	.000	.732
	Roy's Largest Root	2.729	98.238[b]	2.000	72.000	.000	.732
time * Material * Solution	Pillai's Trace	.100	2.001[b]	4.000	72.000	.103	.100
	Wilks' Lambda	.900	2.001[b]	4.000	72.000	.103	.100
	Hotelling's Trace	.111	2.001[b]	4.000	72.000	.103	.100
	Roy's Largest Root	.111	2.001[b]	4.000	72.000	.103	.100

a. Design: Intercept + Material + Solution + Material * Solution
Within Subjects Design: time

b. Exact statistic

Mauchly's Test of Sphericity[a]

Measure: Hardness

Within Subjects Effect	Mauchly's W	Approx. Chi-Square	df	Sig.	Epsilon[b] Greenhouse-Geisser	Huynh-Feldt	Lower-bound
time	1.000	.000	0	.	1.000	1.000	1.000

Tests the null hypothesis that the error covariance matrix of the orthonormalized transformed dependent variables is proportional to an identity matrix.

a. Design: Intercept + Material + Solution + Material * Solution
Within Subjects Design: time

b. May be used to adjust the degrees of freedom for the averaged tests of significance. Corrected tests are displayed in the Tests of Within-Subjects Effects table.

Tests of Within-Subjects Effects

Measure: Hardness

Source		Type III Sum of Squares	df	Mean Square	F	Sig.	Partial Eta Squared
time	Sphericity Assumed	.187	1	.187	356.218	.000	.832
	Greenhouse-Geisser	.187	1.000	.187	356.218	.000	.832
	Huynh-Feldt	.187	1.000	.187	356.218	.000	.832
	Lower-bound	.187	1.000	.187	356.218	.000	.832
time * Material	Sphericity Assumed	.011	2	.006	10.703	.000	.229
	Greenhouse-Geisser	.011	2.000	.006	10.703	.000	.229
	Huynh-Feldt	.011	2.000	.006	10.703	.000	.229
	Lower-bound	.011	2.000	.006	10.703	.000	.229
time * Solution	Sphericity Assumed	.103	2	.052	98.238	.000	.732
	Greenhouse-Geisser	.103	2.000	.052	98.238	.000	.732
	Huynh-Feldt	.103	2.000	.052	98.238	.000	.732
	Lower-bound	.103	2.000	.052	98.238	.000	.732
time * Material * Solution	Sphericity Assumed	.004	4	.001	2.001	.103	.100
	Greenhouse-Geisser	.004	4.000	.001	2.001	.103	.100
	Huynh-Feldt	.004	4.000	.001	2.001	.103	.100
	Lower-bound	.004	4.000	.001	2.001	.103	.100
Error(time)	Sphericity Assumed	.038	72	.001			
	Greenhouse-Geisser	.038	72.000	.001			
	Huynh-Feldt	.038	72.000	.001			
	Lower-bound	.038	72.000	.001			

Tests of Within-Subjects Contrasts

Measure: Hardness

Source	time	Type III Sum of Squares	df	Mean Square	F	Sig.	Partial Eta Squared
time	Linear	.187	1	.187	356.218	.000	.832
time * Material	Linear	.011	2	.006	10.703	.000	.229
time * Solution	Linear	.103	2	.052	98.238	.000	.732
time * Material * Solution	Linear	.004	4	.001	2.001	.103	.100
Error(time)	Linear	.038	72	.001			

Levene's Test of Equality of Error Variances[a]

	F	df1	df2	Sig.
H_day1	5.471	8	72	.000
H_day56	2.596	8	72	.015

Tests the null hypothesis that the error variance of the dependent variable is equal across groups.

a. Design: Intercept + Material + Solution + Material * Solution
Within Subjects Design: time

Tests of Between-Subjects Effects

Measure: Hardness

Transformed Variable: Average

Source	Type III Sum of Squares	df	Mean Square	F	Sig.	Partial Eta Squared
Intercept	5.381	1	5.381	7673.001	.000	.991
Material	.148	2	.074	105.789	.000	.746
Solution	.112	2	.056	80.005	.000	.690
Material * Solution	.007	4	.002	2.663	.039	.129
Error	.050	72	.001			

Estimate marginal means

1. Material

Measure: Hardness

Material	Mean	Std. Error	95% Confidence Interval	
			Lower Bound	Upper Bound
1.0	.141	.004	.133	.148
2.0	.211	.004	.204	.218
3.0	.195	.004	.188	.202

2. Solution

Measure: Hardness

Solution	Mean	Std. Error	95% Confidence Interval	
			Lower Bound	Upper Bound
1.0	.149	.004	.142	.157
2.0	.184	.004	.176	.191
3.0	.214	.004	.207	.221

3. time

Measure: Hardness

time	Mean	Std. Error	95% Confidence Interval	
			Lower Bound	Upper Bound
1	.216	.002	.212	.220
2	.148	.003	.142	.155

4. Material * Solution

Measure: Hardness

Material	Solution	Mean	Std. Error	95% Confidence Interval	
				Lower Bound	Upper Bound
1.0	1.0	.118	.006	.106	.131
	2.0	.130	.006	.118	.143
	3.0	.173	.006	.161	.186
2.0	1.0	.176	.006	.164	.189
	2.0	.219	.006	.206	.231
	3.0	.239	.006	.227	.252
3.0	1.0	.154	.006	.141	.166
	2.0	.202	.006	.189	.214
	3.0	.229	.006	.217	.242

5. Material * time

Measure: Hardness

Material	time	Mean	Std. Error	95% Confidence Interval	
				Lower Bound	Upper Bound
1.0	1	.164	.004	.157	.171
	2	.117	.006	.106	.128
2.0	1	.246	.004	.239	.253
	2	.177	.006	.165	.188
3.0	1	.239	.004	.231	.246
	2	.151	.006	.140	.162

6. Solution * time

Measure: Hardness

Solution	time	Mean	Std. Error	95% Confidence Interval	
				Lower Bound	Upper Bound
1.0	1	.216	.004	.209	.224
	2	.082	.006	.071	.094
2.0	1	.213	.004	.205	.220
	2	.154	.006	.143	.166
3.0	1	.220	.004	.212	.227
	2	.208	.006	.197	.219

7. Material * Solution * time

Measure: Hardness

Material	Solution	time	Mean	Std. Error	95% Confidence Interval	
					Lower Bound	Upper Bound
1.0	1.0	1	.172	.006	.159	.185
		2	.065	.010	.045	.084
	2.0	1	.150	.006	.138	.163
		2	.110	.010	.090	.129
	3.0	1	.170	.006	.157	.182
		2	.177	.010	.157	.196
2.0	1.0	1	.254	.006	.241	.266
		2	.099	.010	.079	.118
	2.0	1	.244	.006	.231	.257
		2	.193	.010	.174	.213
	3.0	1	.240	.006	.227	.253
		2	.238	.010	.219	.258
3.0	1.0	1	.224	.006	.211	.236
		2	.084	.010	.064	.103
	2.0	1	.244	.006	.231	.257
		2	.160	.010	.140	.179
	3.0	1	.249	.006	.236	.262
		2	.210	.010	.190	.229

Repeated mixed Analysis of variance: Elastic modulus

Within-Subjects Factors

Measure: Ei

time	Dependent Variable
1	Ei_day1
2	Ei_day56

Between-Subjects Factors

		N
Material	1.0	27
	2.0	27
	3.0	27
Solution	1.0	27
	2.0	27
	3.0	27

Box's Test of Equality of Covariance Matrices[a]

Box's M	145.636
F	5.450
df1	24
df2	15845.434
Sig.	.000

Tests the null hypothesis that the observed covariance matrices of the dependent variables are equal across groups.

a. Design: Intercept + Material + Solution + Material * Solution
Within Subjects Design: time

Descriptive Statistics

	Material	Solution	Mean	Std. Deviation	N
Ei_day1	1.0	1.0	2.981426671	.1407917350	9
		2.0	2.682472465	.5547572426	9
		3.0	3.043054347	.1235093091	9
		Total	2.902317828	.3622702097	27
	2.0	1.0	3.652647173	.3090189133	9
		2.0	3.370611639	.1003363895	9
		3.0	3.342132725	.1585013626	9
		Total	3.455130513	.2461851270	27
	3.0	1.0	3.616683027	.3378396312	9
		2.0	3.868260331	.4106668420	9
		3.0	3.799238893	.6567355124	9
		Total	3.761394084	.4810573477	27
	Total	1.0	3.416918957	.4114583822	27
		2.0	3.307114812	.6286006980	27
		3.0	3.394808655	.4955205924	27
		Total	3.372947475	.5152855896	81
Ei_day56	1.0	1.0	.8249282060	.2910650685	9
		2.0	1.492946367	.8906764274	9
		3.0	2.513005283	.5386656902	9
		Total	1.610293285	.9272496718	27
	2.0	1.0	1.278409815	.8040921823	9
		2.0	2.684163175	.2225032133	9
		3.0	3.341565722	.2231525426	9
		Total	2.434712904	.9992641627	27
	3.0	1.0	1.403637433	.8796041249	9
		2.0	2.225626448	.3533737763	9
		3.0	2.991928561	.5650047835	9
		Total	2.207064147	.9008354138	27
	Total	1.0	1.168991818	.7261221531	27
		2.0	2.134245330	.7400546284	27
		3.0	2.948833188	.5679884771	27
		Total	2.084023445	.9950131971	81

Multivariate Tests[a]

Effect		Value	F	Hypothesis df	Error df	Sig.	Partial Eta Squared
time	Pillai's Trace	.793	275.222[b]	1.000	72.000	.000	.793
	Wilks' Lambda	.207	275.222[b]	1.000	72.000	.000	.793
	Hotelling's Trace	3.823	275.222[b]	1.000	72.000	.000	.793
	Roy's Largest Root	3.823	275.222[b]	1.000	72.000	.000	.793
time * Material	Pillai's Trace	.099	3.936[b]	2.000	72.000	.024	.099
	Wilks' Lambda	.901	3.936[b]	2.000	72.000	.024	.099
	Hotelling's Trace	.109	3.936[b]	2.000	72.000	.024	.099
	Roy's Largest Root	.109	3.936[b]	2.000	72.000	.024	.099
time * Solution	Pillai's Trace	.558	45.384[b]	2.000	72.000	.000	.558
	Wilks' Lambda	.442	45.384[b]	2.000	72.000	.000	.558
	Hotelling's Trace	1.261	45.384[b]	2.000	72.000	.000	.558
	Roy's Largest Root	1.261	45.384[b]	2.000	72.000	.000	.558
time * Material * Solution	Pillai's Trace	.091	1.801[b]	4.000	72.000	.138	.091
	Wilks' Lambda	.909	1.801[b]	4.000	72.000	.138	.091
	Hotelling's Trace	.100	1.801[b]	4.000	72.000	.138	.091
	Roy's Largest Root	.100	1.801[b]	4.000	72.000	.138	.091

a. Design: Intercept + Material + Solution + Material * Solution
Within Subjects Design: time

b. Exact statistic

Mauchly's Test of Sphericity[a]

Measure: Ei

					Epsilon[b]		
Within Subjects Effect	Mauchly's W	Approx. Chi-Square	df	Sig.	Greenhouse-Geisser	Huynh-Feldt	Lower-bound
time	1.000	.000	0	.	1.000	1.000	1.000

Tests the null hypothesis that the error covariance matrix of the orthonormalized transformed dependent variables is proportional to an identity matrix.

a. Design: Intercept + Material + Solution + Material * Solution
Within Subjects Design: time

b. May be used to adjust the degrees of freedom for the averaged tests of significance. Corrected tests are displayed in the Tests of Within-Subjects Effects table.

Tests of Within-Subjects Effects

Measure: Ei

Source		Type III Sum of Squares	df	Mean Square	F	Sig.	Partial Eta Squared
time	Sphericity Assumed	67.284	1	67.284	275.222	.000	.793
	Greenhouse-Geisser	67.284	1.000	67.284	275.222	.000	.793
	Huynh-Feldt	67.284	1.000	67.284	275.222	.000	.793
	Lower-bound	67.284	1.000	67.284	275.222	.000	.793
time * Material	Sphericity Assumed	1.924	2	.962	3.936	.024	.099
	Greenhouse-Geisser	1.924	2.000	.962	3.936	.024	.099
	Huynh-Feldt	1.924	2.000	.962	3.936	.024	.099
	Lower-bound	1.924	2.000	.962	3.936	.024	.099
time * Solution	Sphericity Assumed	22.190	2	11.095	45.384	.000	.558
	Greenhouse-Geisser	22.190	2.000	11.095	45.384	.000	.558
	Huynh-Feldt	22.190	2.000	11.095	45.384	.000	.558
	Lower-bound	22.190	2.000	11.095	45.384	.000	.558
time * Material * Solution	Sphericity Assumed	1.762	4	.440	1.801	.138	.091
	Greenhouse-Geisser	1.762	4.000	.440	1.801	.138	.091
	Huynh-Feldt	1.762	4.000	.440	1.801	.138	.091
	Lower-bound	1.762	4.000	.440	1.801	.138	.091
Error(time)	Sphericity Assumed	17.602	72	.244			
	Greenhouse-Geisser	17.602	72.000	.244			
	Huynh-Feldt	17.602	72.000	.244			
	Lower-bound	17.602	72.000	.244			

Tests of Within-Subjects Contrasts

Measure: Ei

Source	time	Type III Sum of Squares	df	Mean Square	F	Sig.	Partial Eta Squared
time	Linear	67.284	1	67.284	275.222	.000	.793
time * Material	Linear	1.924	2	.962	3.936	.024	.099
time * Solution	Linear	22.190	2	11.095	45.384	.000	.558
time * Material * Solution	Linear	1.762	4	.440	1.801	.138	.091
Error(time)	Linear	17.602	72	.244			

Levene's Test of Equality of Error Variances[a]

	F	df1	df2	Sig.
Ei_day1	4.078	8	72	.000
Ei_day56	4.343	8	72	.000

Tests the null hypothesis that the error variance of the dependent variable is equal across groups.

a. Design: Intercept + Material + Solution + Material * Solution
Within Subjects Design: time

Tests of Between-Subjects Effects

Measure: Ei

Transformed Variable: Average

Source	Type III Sum of Squares	df	Mean Square	F	Sig.	Partial Eta Squared
Intercept	1206.031	1	1206.031	5121.814	.000	.986
Material	18.101	2	9.050	38.436	.000	.516
Solution	20.860	2	10.430	44.294	.000	.552
Material * Solution	1.053	4	.263	1.118	.355	.058
Error	16.954	72	.235			

Estimated marginal means

1. Material

Measure: Ei

Material	Mean	Std. Error	95% Confidence Interval	
			Lower Bound	Upper Bound
1.0	2.256	.066	2.125	2.388
2.0	2.945	.066	2.813	3.077
3.0	2.984	.066	2.853	3.116

2. Solution

Measure: Ei

Solution	Mean	Std. Error	95% Confidence Interval	
			Lower Bound	Upper Bound
1.0	2.293	.066	2.161	2.425
2.0	2.721	.066	2.589	2.852
3.0	3.172	.066	3.040	3.303

3. time

Measure: Ei

time	Mean	Std. Error	95% Confidence Interval	
			Lower Bound	Upper Bound
1	3.373	.040	3.292	3.453
2	2.084	.066	1.953	2.215

4. Material * Solution

Measure: Ei

Material	Solution	Mean	Std. Error	95% Confidence Interval	
				Lower Bound	Upper Bound
1.0	1.0	1.903	.114	1.675	2.131
	2.0	2.088	.114	1.860	2.316
	3.0	2.778	.114	2.550	3.006
2.0	1.0	2.466	.114	2.238	2.694
	2.0	3.027	.114	2.799	3.255
	3.0	3.342	.114	3.114	3.570
3.0	1.0	2.510	.114	2.282	2.738
	2.0	3.047	.114	2.819	3.275
	3.0	3.396	.114	3.168	3.624

5. Material * time

Measure: Ei

Material	time	Mean	Std. Error	95% Confidence Interval	
				Lower Bound	Upper Bound
1.0	1	2.902	.070	2.763	3.042
	2	1.610	.114	1.384	1.837
2.0	1	3.455	.070	3.316	3.594
	2	2.435	.114	2.208	2.661
3.0	1	3.761	.070	3.622	3.901
	2	2.207	.114	1.981	2.433

6. Solution * time

Measure: Ei

Solution	time	Mean	Std. Error	95% Confidence Interval Lower Bound	Upper Bound
1.0	1	3.417	.070	3.278	3.556
	2	1.169	.114	.943	1.395
2.0	1	3.307	.070	3.168	3.446
	2	2.134	.114	1.908	2.361
3.0	1	3.395	.070	3.255	3.534
	2	2.949	.114	2.723	3.175

7. Material * Solution * time

Measure: Ei

Material	Solution	time	Mean	Std. Error	95% Confidence Interval Lower Bound	Upper Bound
1.0	1.0	1	2.981	.121	2.740	3.223
		2	.825	.197	.433	1.217
	2.0	1	2.682	.121	2.441	2.924
		2	1.493	.197	1.101	1.885
	3.0	1	3.043	.121	2.802	3.284
		2	2.513	.197	2.121	2.905
2.0	1.0	1	3.653	.121	3.411	3.894
		2	1.278	.197	.886	1.670
	2.0	1	3.371	.121	3.129	3.612
		2	2.684	.197	2.292	3.076
	3.0	1	3.342	.121	3.101	3.584
		2	3.342	.197	2.950	3.734
3.0	1.0	1	3.617	.121	3.375	3.858
		2	1.404	.197	1.012	1.796
	2.0	1	3.868	.121	3.627	4.110
		2	2.226	.197	1.834	2.618
	3.0	1	3.799	.121	3.558	4.041
		2	2.992	.197	2.600	3.384

Pair samples t-test: Nanohardness and Elastic modulus

TRX in coffee solution

Paired Samples Statistics

		Mean	N	Std. Deviation	Std. Error Mean
Pair 1	H_day1	.17189	9	.003219	.001073
	H_day56	.06466500	9	.010813245	.003604415
Pair 2	Ei_day1	2.981426671	9	.1407917350	.0469305783
	Ei_day56	.8249282060	9	.2910650685	.0970216895

Paired Samples Correlations

		N	Correlation	Sig.
Pair 1	H_day1 & H_day56	9	.562	.115
Pair 2	Ei_day1 & Ei_day56	9	-.019	.960

Paired Samples Test

		Paired Differences					t	df	Sig. (2-tailed)
		Mean	Std. Deviation	Std. Error Mean	95% Confidence Interval of the Difference Lower	Upper			
Pair 1	H_day1 - H_day56	.107223889	.009388098	.003129366	.100007558	.114440220	34.264	8	.000
Pair 2	Ei_day1 - Ei_day56	2.156498465	.3257858498	.1085952833	1.906077292	2.406919637	19.858	8	.000

BTC in coffee solution

Paired Samples Statistics

		Mean	N	Std. Deviation	Std. Error Mean
Pair 1	H_day1	.25368356	9	.009525759	.003175253
	H_day56	.09865844	9	.033684914	.011228305
Pair 2	Ei_day1	3.652647173	9	.3090189133	.1030063044
	Ei_day56	1.278409815	9	.8040921823	.2680307274

Paired Samples Correlations

		N	Correlation	Sig.
Pair 1	H_day1 & H_day56	9	-.180	.643
Pair 2	Ei_day1 & Ei_day56	9	-.532	.140

Paired Samples Test

		Paired Differences					t	df	Sig. (2-tailed)
		Mean	Std. Deviation	Std. Error Mean	95% Confidence Interval of the Difference Lower	Upper			
Pair 1	H_day1 - H_day56	.155025111	.036616802	.012205601	.126878945	.183171277	12.701	8	.000
Pair 2	Ei_day1 - Ei_day56	2.374237359	1.003327839	.3344426130	1.603011310	3.145463407	7.099	8	.000

TRD in coffee solution

Paired Samples Statistics

		Mean	N	Std. Deviation	Std. Error Mean
Pair 1	H_day1	.22362800	9	.022699990	.007566663
	H_day56	.08381878	9	.048656081	.016218694
Pair 2	Ei_day1	3.616683027	9	.3378396312	.1126132104
	Ei_day56	1.403637433	9	.8796041249	.2932013750

Paired Samples Correlations

		N	Correlation	Sig.
Pair 1	H_day1 & H_day56	9	.189	.626
Pair 2	Ei_day1 & Ei_day56	9	-.369	.328

Paired Samples Test

		Paired Differences					t	df	Sig. (2-tailed)
		Mean	Std. Deviation	Std. Error Mean	95% Confidence Interval of the Difference				
					Lower	Upper			
Pair 1	H_day1 - H_day56	.139809222	.049642126	.016547375	.101650906	.177967538	8.449	8	.000
Pair 2	Ei_day1 - Ei_day56	2.213045595	1.052342294	.3507807647	1.404143701	3.021947488	6.309	8	.000

TRX in distilled water

Paired Samples Statistics

		Mean	N	Std. Deviation	Std. Error Mean
Pair 1	H_day1	.15044	9	.023163	.007721
	H_day56	.10985556	9	.042858236	.014286079
Pair 2	Ei_day1	2.682472465	9	.5547572426	.1849190809
	Ei_day56	1.492946367	9	.8906764274	.2968921425

Paired Samples Correlations

		N	Correlation	Sig.
Pair 1	H_day1 & H_day56	9	.935	.000
Pair 2	Ei_day1 & Ei_day56	9	.891	.001

Paired Samples Test

		Paired Differences					t	df	Sig. (2-tailed)
		Mean	Std. Deviation	Std. Error Mean	95% Confidence Interval of the Difference				
					Lower	Upper			
Pair 1	H_day1 - H_day56	.040588889	.022724111	.007574704	.023121591	.058056187	5.358	8	.001
Pair 2	Ei_day1 - Ei_day56	1.189526099	.4693892113	.1564630704	.8287216112	1.550330586	7.603	8	.000

BTC in distilled water

Paired Samples Statistics

		Mean	N	Std. Deviation	Std. Error Mean
Pair 1	H_day1	.24386422	9	.007713615	.002571205
	H_day56	.19338011	9	.022726140	.007575380
Pair 2	Ei_day1	3.370611639	9	.1003363895	.0334454632
	Ei_day56	2.684163175	9	.2225032133	.0741677378

Paired Samples Correlations

		N	Correlation	Sig.
Pair 1	H_day1 & H_day56	9	-.293	.444
Pair 2	Ei_day1 & Ei_day56	9	-.815	.007

Paired Samples Test

		Paired Differences					t	df	Sig. (2-tailed)
					95% Confidence Interval of the Difference				
		Mean	Std. Deviation	Std. Error Mean	Lower	Upper			
Pair 1	H_day1 - H_day56	.050484111	.026052967	.008684322	.030458028	.070510194	5.813	8	.000
Pair 2	Ei_day1 - Ei_day56	.6864484644	.3098081557	.1032693852	.4483088349	.9245880938	6.647	8	.000

TRD in distilled water

Paired Samples Statistics

		Mean	N	Std. Deviation	Std. Error Mean
Pair 1	H_day1	.24404300	9	.024761069	.008253690
	H_day56	.15969889	9	.026511724	.008837241
Pair 2	Ei_day1	3.868260331	9	.4106668420	.1368889473
	Ei_day56	2.225626448	9	.3533737763	.1177912588

Paired Samples Correlations

		N	Correlation	Sig.
Pair 1	H_day1 & H_day56	9	.119	.761
Pair 2	Ei_day1 & Ei_day56	9	-.141	.717

Paired Samples Test

		Paired Differences					t	df	Sig. (2-tailed)
					95% Confidence Interval of the Difference				
		Mean	Std. Deviation	Std. Error Mean	Lower	Upper			
Pair 1	H_day1 - H_day56	.084344111	.034061003	.011353668	.058162506	.110525716	7.429	8	.000
Pair 2	Ei_day1 - Ei_day56	1.642633883	.5783130982	.1927710327	1.198103084	2.087164682	8.521	8	.000

TRX in dark chamber

Paired Samples Statistics

		Mean	N	Std. Deviation	Std. Error Mean
Pair 1	H_day1	.16967	9	.007616	.002539
	H_day56	.17650122	9	.015525511	.005175170
Pair 2	Ei_day1	3.043054347	9	.1235093091	.0411697697
	Ei_day56	2.513005283	9	.5386656902	.1795552301

Paired Samples Correlations

		N	Correlation	Sig.
Pair 1	H_day1 & H_day56	9	.925	.000
Pair 2	Ei_day1 & Ei_day56	9	.961	.000

Paired Samples Test

		Paired Differences					t	df	Sig. (2-tailed)
		Mean	Std. Deviation	Std. Error Mean	95% Confidence Interval of the Difference				
					Lower	Upper			
Pair 1	H_day1 - H_day56	-.006834556	.008955002	.002985001	-.013717980	.000048869	-2.290	8	.051
Pair 2	Ei_day1 - Ei_day56	.5300490647	.4213545699	.1404515233	.2061672712	.8539308582	3.774	8	.005

BTC in dark chamber

Paired Samples Statistics

		Mean	N	Std. Deviation	Std. Error Mean
Pair 1	H_day1	.24010944	9	.008808334	.002936111
	H_day56	.23804622	9	.012563431	.004187810
Pair 2	Ei_day1	3.342132725	9	.1585013626	.0528337875
	Ei_day56	3.341565722	9	.2231525426	.0743841809

Paired Samples Correlations

		N	Correlation	Sig.
Pair 1	H_day1 & H_day56	9	.673	.047
Pair 2	Ei_day1 & Ei_day56	9	.710	.032

Paired Samples Test

		Paired Differences					t	df	Sig. (2-tailed)
		Mean	Std. Deviation	Std. Error Mean	95% Confidence Interval of the Difference				
					Lower	Upper			
Pair 1	H_day1 - H_day56	.002063222	.009294290	.003098097	-.005081001	.009207446	.666	8	.524
Pair 2	Ei_day1 - Ei_day56	.0005670031	.1571477105	.0523825702	-.120227420	.1213614266	.011	8	.992

TRD in dark chamber

Paired Samples Statistics

		Mean	N	Std. Deviation	Std. Error Mean
Pair 1	H_day1	.24885800	9	.036557034	.012185678
	H_day56	.20973133	9	.025862586	.008620862
Pair 2	Ei_day1	3.799238893	9	.6567355124	.2189118375
	Ei_day56	2.991928561	9	.5650047835	.1883349278

Paired Samples Correlations

		N	Correlation	Sig.
Pair 1	H_day1 & H_day56	9	-.549	.126
Pair 2	Ei_day1 & Ei_day56	9	-.777	.014

Paired Samples Test

		Paired Differences					t	df	Sig. (2-tailed)
		Mean	Std. Deviation	Std. Error Mean	95% Confidence Interval of the Difference Lower	95% Confidence Interval of the Difference Upper			
Pair 1	H_day1 - H_day56	.039126667	.055160231	.018386744	-.003273240	.081526573	2.128	8	.066
Pair 2	Ei_day1 - Ei_day56	.8073103325	1.152065023	.3840216745	-.078245237	1.692865902	2.102	8	.069

One way Analysis of variance: Nanohardness and Elastic modulus

Each material in coffee solution at day 1

ANOVA

		Sum of Squares	df	Mean Square	F	Sig.
Hardness	Between Groups	.031	2	.015	74.981	.000
	Within Groups	.005	24	.000		
	Total	.036	26			
Ei	Between Groups	2.566	2	1.283	16.776	.000
	Within Groups	1.836	24	.076		
	Total	4.402	26			

Multiple Comparisons

Tukey HSD

Dependent Variable	(I) Material	(J) Material	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval Lower Bound	95% Confidence Interval Upper Bound
Hardness	1.0	2.0	-.081794667*	.006757113	.000	-.09866912	-.06492022
		3.0	-.051739111*	.006757113	.000	-.06861356	-.03486466
	2.0	1.0	.081794667*	.006757113	.000	.06492022	.09866912
		3.0	.030055556*	.006757113	.000	.01318110	.04693001
	3.0	1.0	.051739111*	.006757113	.000	.03486466	.06861356
		2.0	-.030055556*	.006757113	.000	-.04693001	-.01318110
Ei	1.0	2.0	-.671220503*	.1303700198	.000	-.996791874	-.345649132
		3.0	-.635256357*	.1303700198	.000	-.960827728	-.309684985
	2.0	1.0	.671220503*	.1303700198	.000	.3456491316	.9967918740
		3.0	.0359641462	.1303700198	.959	-.289607225	.3615355174
	3.0	1.0	.635256357*	.1303700198	.000	.3096849854	.9608277278
		2.0	-.035964146	.1303700198	.959	-.361535517	.2896072250

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

Each material in distilled water at day 1

ANOVA

		Sum of Squares	df	Mean Square	F	Sig.
Hardness	Between Groups	.052	2	.026	65.084	.000
	Within Groups	.010	24	.000		
	Total	.062	26			
Ei	Between Groups	6.382	2	3.191	19.678	.000
	Within Groups	3.892	24	.162		
	Total	10.274	26			

Multiple Comparisons

Tukey HSD

Dependent Variable	(I) Material	(J) Material	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
						Lower Bound	Upper Bound
Hardness	1.0	2.0	-.093419778*	.009463920	.000	-.11705390	-.06978565
		3.0	-.093598556*	.009463920	.000	-.11723268	-.06996443
	2.0	1.0	.093419778*	.009463920	.000	.06978565	.11705390
		3.0	-.000178778	.009463920	1.000	-.02381290	.02345535
	3.0	1.0	.093598556*	.009463920	.000	.06996443	.11723268
		2.0	.000178778	.009463920	1.000	-.02345535	.02381290
Ei	1.0	2.0	-.688139174*	.1898284302	.004	-1.16219527	-.214083080
		3.0	-1.18578787*	.1898284302	.000	-1.65984396	-.711731772
	2.0	1.0	.688139174*	.1898284302	.004	.2140830802	1.162195268
		3.0	-.497648692*	.1898284302	.038	-.971704786	-.023592598
	3.0	1.0	1.18578787*	.1898284302	.000	.7117317722	1.659843960
		2.0	.497648692*	.1898284302	.038	.0235925982	.9717047858

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

Each material in dark chamber at day 1

ANOVA

		Sum of Squares	df	Mean Square	F	Sig.
Hardness	Between Groups	.034	2	.017	34.575	.000
	Within Groups	.012	24	.000		
	Total	.046	26			
Ei	Between Groups	2.611	2	1.305	8.302	.002
	Within Groups	3.773	24	.157		
	Total	6.384	26			

Multiple Comparisons

Tukey HSD

Dependent Variable	(I) Material	(J) Material	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
						Lower Bound	Upper Bound
Hardness	1.0	2.0	-.070442778*	.010442092	.000	-.09651968	-.04436587
		3.0	-.079191333*	.010442092	.000	-.10526824	-.05311443
	2.0	1.0	.070442778*	.010442092	.000	.04436587	.09651968
		3.0	-.008748556	.010442092	.684	-.03482546	.01732835
	3.0	1.0	.079191333*	.010442092	.000	.05311443	.10526824
		2.0	.008748556	.010442092	.684	-.01732835	.03482546
Ei	1.0	2.0	-.299078378	.1869202176	.265	-.765871830	.1677150741
		3.0	-.756184546*	.1869202176	.001	-1.22297800	-.289391094
	2.0	1.0	.2990783777	.1869202176	.265	-.167715074	.7658718296
		3.0	-.457106168	.1869202176	.056	-.923899620	.0096872835
	3.0	1.0	.756184546*	.1869202176	.001	.2893910942	1.222977998
		2.0	.4571061683	.1869202176	.056	-.009687284	.9238996201

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

Each material in coffee solution at day 56

ANOVA

		Sum of Squares	df	Mean Square	F	Sig.
Hardness	Between Groups	.005	2	.003	2.167	.136
	Within Groups	.029	24	.001		
	Total	.034	26			
Ei	Between Groups	1.669	2	.834	1.663	.211
	Within Groups	12.040	24	.502		
	Total	13.709	26			

Multiple Comparisons

Tukey HSD

Dependent Variable	(I) Material	(J) Material	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval Lower Bound	Upper Bound
Hardness	1.0	2.0	-.033993444	.016372999	.116	-.07488152	.00689464
		3.0	-.019153778	.016372999	.482	-.06004186	.02173430
	2.0	1.0	.033993444	.016372999	.116	-.00689464	.07488152
		3.0	.014839667	.016372999	.642	-.02604841	.05572775
	3.0	1.0	.019153778	.016372999	.482	-.02173430	.06004186
		2.0	-.014839667	.016372999	.642	-.05572775	.02604841
Ei	1.0	2.0	-.453481609	.3338869323	.378	-1.28729314	.3803299267
		3.0	-.578709227	.3338869323	.214	-1.41252076	.2551023089
	2.0	1.0	.4534816089	.3338869323	.378	-.380329927	1.287293144
		3.0	-.125227618	.3338869323	.926	-.959039153	.7085839178
	3.0	1.0	.5787092267	.3338869323	.214	-.255102309	1.412520762
		2.0	.1252276178	.3338869323	.926	-.708583918	.9590391534

Each material in distilled water at day 56

ANOVA

		Sum of Squares	df	Mean Square	F	Sig.
Hardness	Between Groups	.032	2	.016	15.601	.000
	Within Groups	.024	24	.001		
	Total	.056	26			
Ei	Between Groups	6.498	2	3.249	10.073	.001
	Within Groups	7.741	24	.323		
	Total	14.240	26			

Multiple Comparisons

Tukey HSD

Dependent Variable	(I) Material	(J) Material	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval Lower Bound	Upper Bound
Hardness	1.0	2.0	-.083524556*	.015046046	.000	-.12109885	-.04595026
		3.0	-.049843333*	.015046046	.008	-.08741763	-.01226904
	2.0	1.0	.083524556*	.015046046	.000	.04595026	.12109885
		3.0	.033681222	.015046046	.085	-.00389308	.07125552
	3.0	1.0	.049843333*	.015046046	.008	.01226904	.08741763
		2.0	-.033681222	.015046046	.085	-.07125552	.00389308
Ei	1.0	2.0	-1.19121681*	.2677319284	.000	-1.85982030	-.522613312
		3.0	-.732680082*	.2677319284	.030	-1.40128358	-.064076585
	2.0	1.0	1.19121681*	.2677319284	.000	.5226133118	1.859820305
		3.0	.4585367267	.2677319284	.221	-.210066770	1.127140223
	3.0	1.0	.732680082*	.2677319284	.030	.0640765852	1.401283578
		2.0	-.458536727	.2677319284	.221	-1.12714022	.2100667697

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

Each material in dark chamber at day 56

ANOVA

		Sum of Squares	df	Mean Square	F	Sig.
Hardness	Between Groups	.017	2	.009	23.996	.000
	Within Groups	.009	24	.000		
	Total	.026	26			
Ei	Between Groups	3.114	2	1.557	7.087	.004
	Within Groups	5.274	24	.220		
	Total	8.388	26			

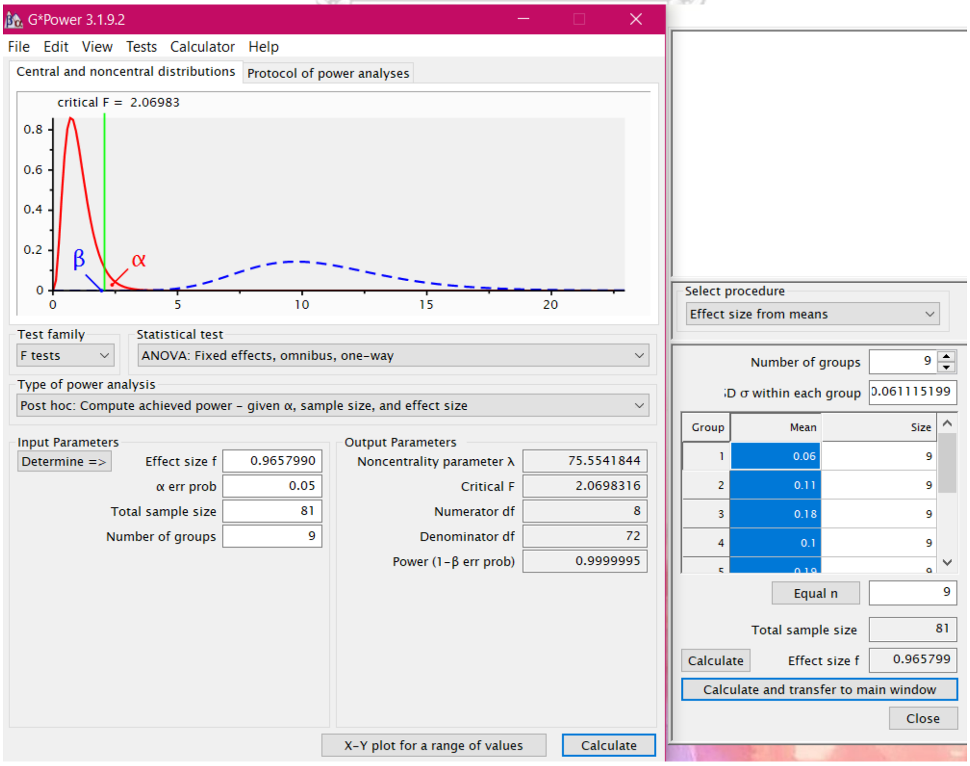
Multiple Comparisons

Tukey HSD

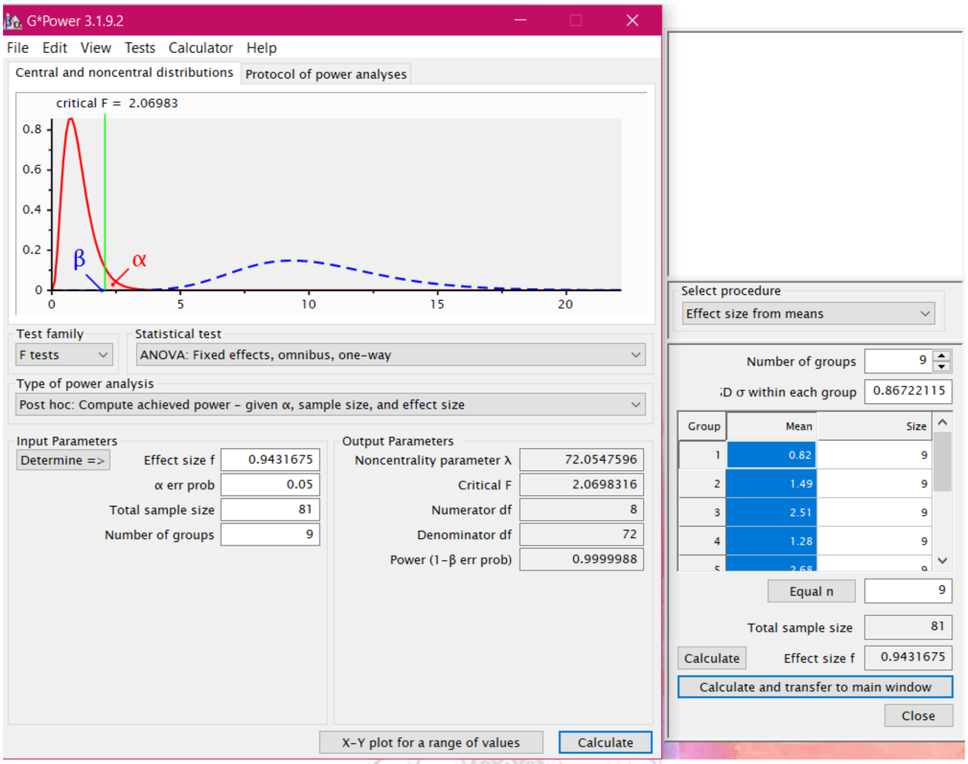
Dependent Variable	(I) Material	(J) Material	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
						Lower Bound	Upper Bound
Hardness	1.0	2.0	-.061545000*	.008893421	.000	-.08375443	-.03933557
		3.0	-.033230111*	.008893421	.003	-.05543954	-.01102069
	2.0	1.0	.061545000*	.008893421	.000	.03933557	.08375443
		3.0	.028314889*	.008893421	.011	.00610546	.05052431
	3.0	1.0	.033230111*	.008893421	.003	.01102069	.05543954
		2.0	-.028314889*	.008893421	.011	-.05052431	-.00610546
Ei	1.0	2.0	-.828560439*	.2209722940	.003	-1.38039170	-.276729176
		3.0	-.478923278	.2209722940	.098	-1.03075454	.0729079853
	2.0	1.0	.828560439*	.2209722940	.003	.2767291758	1.380391703
		3.0	.3496371610	.2209722940	.272	-.202194103	.9014684246
	3.0	1.0	.4789232783	.2209722940	.098	-.072907985	1.030754542
		2.0	-.349637161	.2209722940	.272	-.901468425	.2021941025

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

Power analysis of nanohardness outcome variable (Power = 0.99)



Power analysis of elastic modulus outcome variable (Power = 0.99)



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