

Physical Properties Effect of Dry-Heat and Microwave-Cured Acrylic Resins depending on the Irradiation-Induced Changes

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유도광선변화에 따른 건식중합과 마이크로파중합 아크릴레진의 물리적 성질영향

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Abstract The purpose of this study was to research the property change of acrylic resins depending on the induced-beam change and research the improved physical property of dry-heat and microwave-cured dental place acrylic resin in order to develop the acrylic resins with the optimum characteristic. As a result of observing flexural rigidity, hardness and color difference, the dry-heat-cured specimens of Vertex RS and Paladent 20 showed ideal property at 5, 15, and 25 kGy irradiation. The microwave-cured specimens of Vertex RS and Paladent 20 showed ideal property at 5 kGy irradiation. The correlation analysis showed a positive correlation among ARD, flexural rigidity (0.418), E coefficient (0.675) and Barcol hardness (0.588). The radiation cure technology is helpful for relieving the contamination caused by the manufacture of polymer composite. It can significantly contribute to the fusion of ultra violet cure technology and nano technology and the improvement of mechanical property without giving effect to the workability of polymer.

요약 이 연구는 아크릴 수지의 유도광선변화에 따른 속성 변화를 연구하여 최적의 특성을 가진 아크릴 수지를 개발할 수 있도록 건열 및 마이크로파 경화 의치상 아크릴 수지의 향상된 물성에 대한 연구이다. 굽힘강도와 경도, 색차를 관찰한 결과 Vertex RS 및 Paladent 20는 건식 열 경화 시편 5, 15, 25 kGy의 조사에 이상적인 속성을 보였다. 마이크로 웨이브 경화에서는 5 kGy의 조사에서 이상적인 속성을 보였다. 상관관계 분석은 ARD와 굴곡 강도 (0.418), E 계수 (0.675), 및 바콜 경도 (0.588) 사이의 긍정적인 상관관계를 보여 주었다. 방사선 경화형 기술은 고분자 복합 재료의 제작으로 인한 오염 문제를 완화하는 데 도움이 될 수 있으며 고분자의 가공성에 영향을 주지 않고 자외선 경화 기술과 나노 기술 융합 및 기계적 특성의 향상에 크게 기여할 수 있다.

Keywords : Acrylic resins, Dry-heat cured, Microwave-cured, Irradiation

1. Introduction

Currently, acrylic resin is widely used as a denture base material owing to its optimal strength, simple

production method, aesthetic appeal, and dimensional stability[1-3]. However, due to its insufficient shear strength, fatigue resistance, and impact strength, it is vulnerable to fractures[4,5]. To ensure a perfect

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aesthetic harmony between the denture base and the oral cavity, the color of the resin should be identical to that of natural gum or perfectly transparent to allow for the color of natural gum to be exposed. However, the oral-cavity environment, which is under high stress levels owing to effect of saliva and food-induced rapid temperature and pH changes, significantly affects the quality/stability of resin substrates, and thus, poses a serious challenge to the maintenance of its color[6-8]. Although numerous studies have been conducted to reduce fracture and enhance the flexural strength of acrylic resins during its polymerization using various elastic materials such as aramid[9,10], carbon[11,12], glass fiber[13,14] and polyethylene[15] these studies had limitations regarding homogeneity in material distribution and color stability. When fabricating acrylic resins, care should be taken to ensure its resistance against discoloration or staining in addition to its optimal mechanical strength[16].

The most widely employed method for acrylic resin fabrication is heat curing, in which the polymers and monomers are mixed in a flask and then polymerized at a pre-determined temperature using a water bath[17]. The compression-molding technique, one of the heat curing methods, uses dry heating curing in which the denture base acrylic resin is injected under a certain amount of pressure and polymerized on a hot plate under constant and continuous pressure[18]. The following are different polymerization techniques which can be employed: copolymerization[19], where the polymer arrays are modified using thermocycling or other processes and the polymers have a chain structure, and cross-linking polymerization[20], where covalent bonds are formed by cross-linking of homogeneous polymers. Moreover, visible-light polymerization, pourable-auto curing, and, of late, microwave irradiation curing have been used to facilitate the process of making denture. In microwave-irradiation curing, microwaves are used to polymerize resins placed in their respective flasks. The microwave curing method was first introduced

clinically by Nishi in 1968, and it enables a rapid curing by simultaneously heating the core and margins of the denture base acrylic resin (throughout its mass) via the dielectric loss produced in it, using the microwave energy generated by a magnetron oscillator as the heat source[21].

Because irradiated materials possess thermal stability, flame retardancy, abrasion resistance, and mechanical strength, which improves their overall physical properties, the radiation curing of polymeric materials has a wide variety of applications[22]. Radiation-mediated processing is an economical and eco-friendly method capable of improving the physical, chemical, and mechanical properties of polymeric materials[23]. Radiation technology is a highly sophisticated technology with unlimited potential that is yet to be explored in many fields, which requires the development of various application techniques[24].

This study aims to develop acrylic resins with optimal properties by studying the irradiation induced property changes of Vertex RS (Vertex, the Netherlands) and Paladent 20 (Heraeus Kulzer, Germany), two types of dry-heat and microwave-cured denture base acrylic resins.

2. Materials And Methods

2.1 Experimental materials

Vertex RS (Vertex, Netherlands) and Paladent 20 (Heraeus Kulzer, Germany), two types of denture base acrylic resins, were used as experimental materials. The specimens obtained were polymerized using dry-heat and microwave-curing methods (Table 1).

The specimens were prepared and classified into five experimental groups to measure the flexural strength, E modulus, Barcol hardness, Vickers hardness, and difference in color tone with varying absorbed radiation dose (ARD)(Fig. 1, Fig. 2).

Table 1. Materials and curing methods

Method	Material/Product	Powder/Liquid ratio	Batch NO(P/L)	Polymerization Procedure
Dry-Heat	Vertex RS	2.15 g / 1 mL	YX111P04/ YG505L01	Air-pressurized at 7bar, heat curing at 72 °C for 10min, and heating 100 °C for 30min and 120 °C for 30min.
	Paladent 20	10 g / 4 mL	010521/ 012326	
Microwave	Vertex RS	2.15 g / 1 mL	YX111P04/ YG505L01	Heating 350W by introduction range for 15 min.
	Paladent 20	10 g / 4 mL	010521/ 012326	

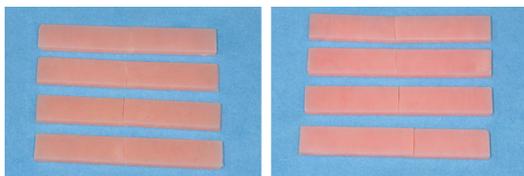


Fig. 1. Dry of denture specimens(Paladent and Vertex).

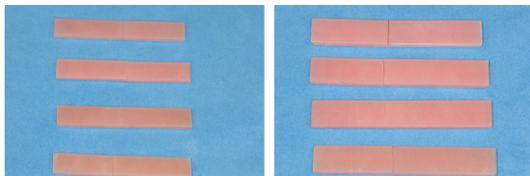


Fig. 2. Microwave of denture specimens(Paladent and Vertex).

2.2 Curing methods and specimen preparation procedure

Two types of dry-heat-cured resins, Vertex RS (powder/solution = 2.15 g / 1 mL) and Paladent 20 (powder/solution = 10 g / 4 mL), were mixed for 30 s. After 5 min, each of the solution in the cylinder was injected into the flask under a constant pressure of 7 bars. After 10 min, mooring was performed three times (in the order of 90 min at 72 °C, 30 min at 100 °C, and 30 min at 120 °C), and the flask was then cooled to room temperature.

The deflasked specimens were ground and polished with #800 and #1200 SiC grit papers down to the exact size of ISO 20795-1 (3.0 x 10 x 65 mm). After the

fabrication, the specimens were stored at 37 °C for 48 h in sealed containers filled with distilled water.

The two types of microwave-cured resin, Vertex RS (powder/solution = 2.15 g / 1 mL) and Paladent 20 (powder/solution = 10 g / 4 mL), were dissolved for 30 s each. After injecting each of the mixtures into its own pre-cast flask, polymerization for 15 min at 350 W output was carried out in a microwave oven (KR-A202B, Daewoo Electronics, Seoul, South Korea). A ceramic plate placed at the bottom of the lower flask diffused the heat upwards, and the glass beads at the top of the upper flask were mixed with plaster and buried, which prevents heat loss from the flask. A total of 28 deflasked specimens were ground and polished with grit papers (#800 and #1200 SiC) down to the SCI-specified size (65 x 15 x 3 mm). Finally, the prepared specimens were stored at 37 °C for 48 h in sealed containers filled with distilled water.

2.3 Test

2.3.1 Irradiation

The irradiation was performed in the ranges of 5, 15, and 25 kGy using a radiation source (60 Co), which was sealed in a double-walled stainless steel capsule, using a JS-10000 Hanging Tote Irradiator(MDS-Nordion, Canada) installed in Soyagreentec, South Korea. Above 25 kGy irradiation, a significant change in color tone was observed by a rough examination, and below 5 kGy irradiation, no changes in hardness, strength, and elasticity were observed. Therefore, the irradiations below 5 kGy and above 25 kGy were excluded from the experiments.

2.3.2 Measurement of flexural strength

The flexural strength was measured using a material testing machine (Instron 4432, Instron, USA) at a cross-head speed of 5.0 mm/min and a point-to-point distance of 50 mm. The maximum flexural strength (FS, MPa) was calculated from the load-displacement curves, which was obtained from the test data using Bluehill 2 (Instron). Flexural strength (a) can be

calculated by using the formula given below, and the data was automatically computed by the Bluehill 2 (Instron) program.

Uniaxial flexural strength (UFS) was calculated from the maximum loads of the specimens by using the formula given below:

$$FS=3PL/2bd^2 \quad (a)$$

where P is the maximal load, L is 50 mm (the length of the support span), (b) is the breadth of specimen, and d is the height of specimen.

2.3.3 Measurement of E modulus

The flexural modulus of elasticity (E, GPa) was calculated from the load-displacement curves, which was obtained from the flexural strength measurement data using Bluehill 2 (Instron). The modulus of elasticity (E modulus) can be calculated by using the formula given below, and the data was automatically computed by the Bluehill 2 (Instron) program:

$$E=L3m/4bd^3 \quad (b)$$

where L is the length of the support span (mm), m is the slope of the modulus line (N/mm), b is the width of specimen (mm), and d is the height of the specimen (mm).

2.3.4 Measurement of Barcol hardness

Digital Barcol hardness tester (HHP-2001, Bareiss, Oberdischingen, Germany) has a metal tipped-indenter having a 0.154 mm diameter and was placed 0.76 mm from the material surface, with a constant load of 70 - 90 N applied to it by a finger. The pressure applied at the instant of indentation is recorded by a sensor on a scale of 0 - 100. The measured distance from the specimen was set to be at least 1 mm, and the mean value was obtained from three measurements per specimen.

2.3.5 Measurement of Vickers hardness

The mean values of Vickers hardness were obtained from the hardness tests using a Vickers hardness testing machine (HM-221, Mitutoyo, Tokyo, Japan)

performed at three points for each specimen by applying an indentation load of 300 g for 30 s at 10-mm intervals from the center of the width. The Vickers hardness was calculated by using the formula given below:

$$HV=1.8544F/d^2 \quad (c)$$

where F is the indentation load applied (300 g) and d is the length of the indent diagonal left on the specimen surface (mm).

2.3.6 Measurement of color stability

The reflected color of each specimen was measured using a portable spectrophotometer (Spectrophotometer CM-2600, Konica Minolta, Japan). The tristimulus values in the CIE standard colorimetric system were obtained from the mean values of the measurements (in duplicates). The conditions of the measurements were as follows: (i) 10 nm wavelength interval, (ii) 3 mm measurement aperture, (iii) specular light removal method using specular component excluded (SCE), and (iv) D65 as the standard light source. The data analyses was performed using a color-control computer program (CM-S100w SpectraMagic NX, Konica Minolta, Japan).

The color stability increases as the color difference decreases. In order to estimate the difference in the color tone for each specimen, CIE $L^*a^*b^*$ (CIELAB) color differences were calculated from the mean values of L, a, and b, which were measured in triplicates by using the following formula:

$$\Delta E^* = \text{where } L \text{ is the luminance, } a = +\text{red} - \text{green}, \\ b = +\text{yellow} - \text{blue}. \quad (d)$$

2.4 Statistical processing

The multi-way analysis of variance (ANOVA) was used to analyze the ARD-dependent differences between the dry-heat and microwave-cured denture base acrylic resins prepared from Vertex RS (Vertex Rapid Simplified, the Netherlands) and Paladent 20 (Heraeus Kulzer, Germany) with respect to flexural strength, E modulus, Vickers hardness, Barcol

hardness, and color stability. A correlation analysis was used to analyze the correlations between flexural strength, E modulus, Vickers hardness, Barcol hardness, and color stability.

3. Results

3.1 Flexural strength and E modulus

The E modulus of the Vertex RS dry-heat-cured denture base acrylic resin was found to be the highest (2.85 ± 0.05) when it was irradiated at 25 kGy. All the irradiated specimens, at the three experimental doses of 5, 15, and 25 kGy, showed higher E modulus values compared to the unirradiated specimens ($p < 0.001$). Similarly, the E modulus of the Paladent 20 dry-heat-cured denture base acrylic resin was the highest (2.92 ± 0.09) when it was irradiated at 25 kGy. All the specimens irradiated at 5, 15, and 25 kGy showed higher E modulus values compared to the unirradiated specimens ($p < 0.01$). For both Vertex RS and Paladent 20 dry-heat-cured denture base acrylic resins, the ARD values that improved the E modulus were 5, 15, and 25 kGy (Fig 3).

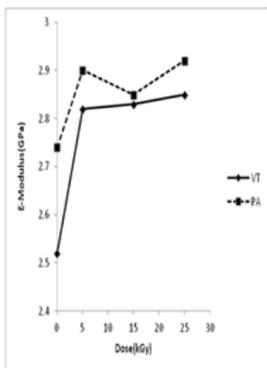


Fig. 3. ARD-dependent changes in E modulus of dry denture materials.

The E modulus of the Vertex RS microwave-cured denture base acrylic resins was highest (3.04 ± 0.19) at 25 kGy. The specimens irradiated at 5, 15, and 25 kGy showed higher E modulus values compared to the

unirradiated specimens ($p < 0.001$). Similarly, the E modulus of the Paladent 20 microwave-cured denture base acrylic resin was the highest (2.88 ± 0.07) when it was irradiated at 25 kGy. The specimens that were irradiated at 15 and 25 kGy showed higher E modulus values compared to the unirradiated specimens ($p < 0.001$). (Fig.4).

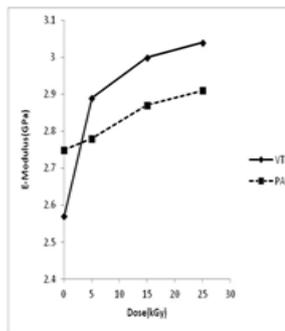


Fig. 4. ARD-dependent changes in E modulus of microwave denture materials.

3.2 Barcol hardness and Vickers hardness

The Barcol hardness of the Vertex RS dry-heat-cured denture base acrylic resins was the highest (43.18 ± 1.08) when they were irradiated at 25 kGy. All the specimens that were irradiated at 5, 15 and 25 kGy showed higher Barcol hardness values compared to the unirradiated specimens ($p < 0.05$).

In contrast, for the Paladent 20 dry-heat-cured denture base acrylic resins, no significant change in Barcol hardness was observed after the irradiation. The ARD values that improved the Barcol hardness were 5, 15, and 25 kGy for the Vertex RS dry-heat-cured denture base acrylic resins, while no correlation was observed for the Paladent 20 dry-heat-cured denture base acrylic resins (Fig 5).

The Barcol hardness of the Vertex RS microwave-cured denture base acrylic resins was the highest (44.31 ± 0.79) when they were irradiated at 5 kGy ($p < 0.001$). The Barcol hardness of the Paladent 20 microwave-cured denture base acrylic resins was the highest (45.81 ± 0.99) when they were irradiated at 15 kGy. All the specimens that were irradiated at 5, 15,

and 25 kGy showed higher Barcol hardness compared to the unirradiated specimen ($p < 0.001$) (Fig. 6).

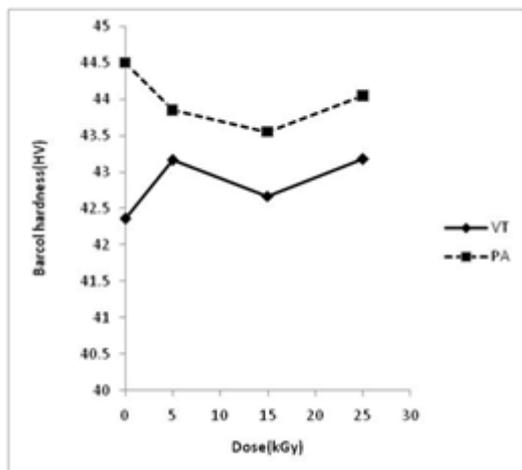


Fig. 5. ARD-dependent changes in Barcol hardness of dry-heat denture materials.

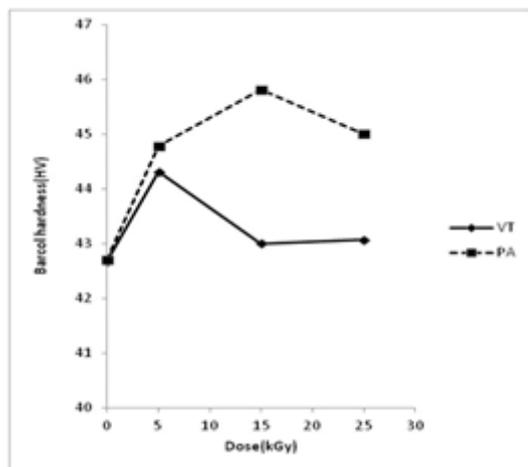


Fig. 6. ARD-dependent changes in Barcol hardness of microwave denture materials.

3.3 Color stability

The color stability of the Vertex RS microwave-cured denture base acrylic resins was satisfactory, showing little color difference (1.75 ± 0.49), when they were irradiated at 5 kGy ($p < 0.001$). The Paladent 20 microwave-cured denture base acrylic resins did not show any statistically significant differences in color stability. The irradiated specimens of both Vertex RS

and Paladent 20 microwave-cured denture base acrylic resins exhibited reduced color stability; however, they were within the clinically permissible range depending on the dose, and did not exhibit significant color differences when analyzed against the standards set by the American Dental Association. Therefore, they can be safely used.

3.4 Specimens' correlations with strength, elasticity, hardness, color stability, and ARD

The correlation analysis to validate this result revealed a positive correlation between the ARD and E modulus (0.572). For the DHP, the E modulus could be enhanced as per the ISO standards at 5, 15, and 25 kGy irradiation, also within the clinically permissible range for the change in color tone to satisfy aesthetic appeal. The correlation analysis to validate this result revealed a positive correlation between the ARD and E modulus (0.387). In the case of MWV, the elasticity and hardness could be improved while maintaining the level of strength required by the ISO standards at an ARD of 5 and 15 kGy, maintaining the change in color tone within the clinically permissible range, thus satisfying aesthetic demands.

The correlation analysis to validate this result showed positive correlations between the ARD and flexural strength (0.381), E modulus (0.787), and color stability (0.830). With regard to the MWP, the E modulus and Barcol hardness could be improved while maintaining the level of strength required by the ISO standards at 5 kGy irradiation, also satisfying aesthetic demands by maintaining the change in color tone within the clinically permissible range.

The correlation analysis to validate this result showed positive correlations between the ARD and flexural strength (0.418), E modulus (0.675), and Barcol hardness (0.588).

Table 2. Correlations with ARD, strength, elasticity, hardness, and color stability

Classification	ARD	Fracture strength	E-Modulus	Barcol hardness	Vickers hardness	Color stability
VT	ARD	1				
	Fracture strength	0.234	1			
	E-Modulus	0.572**	0.284	1		
	Barcol hardness	0.416	-0.367*	0.191	1	
	Vickers hardness	-0.082	-0.139	0.206	0.064	1
	Color stability	0.210	-0.183	-0.172	-0.053	0.075
PA	ARD	1				
	Fracture strength	-0.001	1			
	E-Modulus	0.387**	-0.150	1		
	Barcol hardness	-0.103	-0.133	-0.291	1	
	Vickers hardness	-0.162	-0.156	-0.075	-0.010	1
	Color stability	0.097	0.097	-0.015	-0.266	0.251
VT	ARD	1				
	Fracture strength	0.381*	1			
	E-Modulus	0.787**	0.321	1		
	Barcol hardness	-0.104	-0.110	0.147	1	
	Vickers hardness	-0.182	0.035	-0.250	-0.236	1
	Color stability	0.830**	0.313	0.517**	-0.400**	0.031
PT	ARD	1				
	Fracture strength	0.418*	1			
	E-Modulus	0.675**	0.264	1		
	Barcol hardness	0.588**	0.162	0.546**	1	
	Vickers hardness	-0.236	0.181	-0.248	-0.195	1
	Color stability	0.306	0.165	0.167	0.223	-0.175

The fusion of radiation-curing technology and nanotechnology without affecting the processability of high polymers will significantly contribute to the improvement of mechanical properties such as strength and modulus and suppress the emission of ecologically harmful substances during the process[22].

4. Discussion

The irradiation experiments were performed on the Vertex RS and Paladent 20 heat-cured resins, prepared by using both dry heat and microwave energy, in order to find out the irradiation-induced property changes by comparing the flexural strength, E modulus, Vickers hardness, Barcol hardness, and color stability of the following four types of resin: DHV (dry-heat-cured Vertex RS), DHP (dryheat-cured Paladent 20), MWV (microwave-cured Vertex RS), and MWP (microwave-cured Paladent20). Two types of heat-cured resins using both dry heat and microwave energy (DHV, DHP, MWV, and MWP) were irradiated with varying ARD, and the changes in the properties were compared by measuring their flexural strength, E modulus, Vickers hardness, Barcol hardness, and color stability.

The E modulus and Barcol hardness of the DHV were improved by irradiating at 5, 15, and 25 kGy. Therefore, in order to enhance the E modulus and Barcol hardness of DHV without decreasing its color stability, it is desirable to irradiate it at 5, 15, and 25 kGy. The E modulus of DHP could be improved by irradiating at 5, 15, and 25 kGy. However, the irradiation did not influence its Barcol hardness. Therefore, in order to enhance the E modulus of DHP, it is desirable to irradiate it at 5, 15, and 25 kGy. The E modulus of MWV was improved by irradiating at 15 and 25 kGy. Moreover, its Barcol hardness was enhanced by irradiating at 5 kGy. The color stability was found to be excellent when irradiated at 5 and 15 kGy by demonstrating a negligible change in color tone.

Therefore, in order to enhance the E modulus and Barcol hardness of the MWV while maintaining its color stability, it is desirable to irradiate it at 5 and 15 kGy. In the case of MWP, the flexural strength and E modulus could be simultaneously improved at an ARD of 5, 15, and 25 kGy. The Barcol hardness was also enhanced under these irradiation conditions, especially

at 15 kGy. With respect to color stability, no statistically significant differences were observed, and the specimens irradiated at 5 kGy demonstrated color stability within the clinically permissible range. Therefore, a 5 kGy irradiation is the best recommendation for the enhancement of the MWP's flexural strength, E modulus, and Barcol Hardness while maintaining the desired color stability.

According to the ISO standards (ISO 1567), the flexural strength of heat-cured resins should reach at least 65 MPa, in water 80 MPa[25]. The post-irradiation flexural strengths of the DHV and DHP used in the present study were 82.31 - 93.11 and 98.83 - 104.04 MPa, respectively, thus satisfying the ISO standards. Both the MWV and MWP also satisfied the ISO standards, by showing 92.44 - 101.22 and 98.98 - 103.36 MPa, respectively.

With regard to color stability, the American Dental Association specified a E value of 2 as a reference value for distinguishable difference in color tone, 26 and Eldiwanly indicated a E value of 3.3 as being the maximum clinically permissible color tone difference[27]. O'Brien et al. specified a E value of 3.7 as the minimum level clearly distinguishable by a rough examination[28]. Both the DHV and DHP were within the clinically permissible range of color tone difference. The MWV and MWP were within the clinically permissible range of color tone difference when irradiated at 5 kGy, which is at the same time the ideal ARD for improving all the physical properties. MDS Nordion (Ottawa, Canada) states that the permissible radiation dose for polymethylmethacrylate materials is 100 kGy, and that shades of yellow may partially appear in the range of 20 - 40 kGy irradiation.²⁹ In fact, in the preliminary experiments of our study, clinically impermissible color tone changes were observed at the ranges exceeding 25 kGy irradiation, and therefore, we limited the maximum dose to 25 kGy irradiation.

Therefore, a significant improvement in the elasticity and hardness of DHV was achieved at 5, 15, and 25

kGy irradiation while maintaining the level of strength required by the ISO standards, thereby also satisfying the clinically permissible range for the change in color tone, which is an important aesthetic aspect.

The correlation analysis to validate this result revealed a positive correlation between the ARD and E modulus (0.572). For the DHP, the E modulus could be enhanced as per the ISO standards at 5, 15, and 25 kGy irradiation, also within the clinically permissible range for the change in color tone to satisfy aesthetic appeal. The correlation analysis to validate this result revealed a positive correlation between the ARD and E modulus (0.387). In the case of MWV, the elasticity and hardness could be improved while maintaining the level of strength required by the ISO standards at an ARD of 5 and 15 kGy, maintaining the change in color tone within the clinically permissible range, thus satisfying aesthetic demands. The correlation analysis to validate this result showed positive correlations between the ARD and flexural strength (0.381), E modulus (0.787), and color stability (0.830). With regard to the MWP, the E modulus and Barcol hardness could be improved while maintaining the level of strength required by the ISO standards at 5 kGy irradiation, also satisfying aesthetic demands by maintaining the change in color tone within the clinically permissible range. The correlation analysis to validate this result showed positive correlations between the ARD and flexural strength (0.418), E modulus (0.675), and Barcol hardness (0.588).

Furthermore, radiation-curing technology can help to alleviate the pollution problems caused by the fabrication of polymer composite materials. The fusion of radiation-curing technology and nanotechnology without affecting the processability of high polymers will significantly contribute to the improvement of mechanical properties such as strength and modulus and suppress the emission of ecologically harmful substances during the process[22].

5. Conclusion

In this study, dry-heat and microwave-cured denture base acrylic resins were irradiated, resulting in enhanced physical properties (elasticity and hardness). Despite the known effect of irradiation on enhancing the abrasion resistance of ultrahigh-molecular-weight polyethylene, proper research has not yet been conducted on the degree of cross-linking, absorbed radiation dose, heat-treatment conditions, etc.

The conclusions are as follows. First, the dry-heat-cured specimens of Vertex RS and Paladent 20 showed ideal property changes at 5, 15, and 25 kGy irradiation. Two, the microwave-cured specimens of Vertex RS and Paladent 20 showed ideal property changes at 5 kGy irradiation. Because the radiation-mediated fabrication of polymer composite materials is able to produce materials with excellent physical properties, it is expected to be established as a 21st-century eco-friendly technology.

In this study, given the importance of adequate irradiation dose, determination of optimum property changes in the Vertex RS and Paladent 20 heat-cured resins with respect to the ARD was attempted. And it is necessary to conduct further follow-up studies that would apply the results of this study in clinical settings and prove the effects of radiation technology from various practical perspectives.

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<Research Interests>

Dental material, Health education