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EVALUATION OF IN-VITRO AGING PROCEDURES IN DENTISTRY: A TRADITIONAL REVIEW

DİŞ HEKİMLİĞİNDE UYGULANAN İN-VITRO YAŞLANDIRMA YÖNTEMLERİNİN DEĞERLENDİRİLMESİ: GELENEKSEL BİR DERLEME

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Abstract

The longevity of dental restorative materials is a critical factor for successful clinical outcomes. Various in-vitro aging protocols have been developed to simulate oral environmental conditions such as thermal fluctuations, pH variations, enzymatic activity, and mechanical forces. Common methods include water storage, sodium hypochlorite (NaOCl) immersion, pH cycling, thermal cycling, and chewing simulation. Each technique provides insights into specific aspects of material durability, including hydrolytic degradation, chemical resistance, thermal stability, and mechanical wear. However, existing protocols often fall short of fully replicating the complex and dynamic intraoral environment. This review analyzes the strengths and limitations of current in-vitro aging procedures and underscores the need for the development of more comprehensive and clinically relevant simulation methods.

Keywords: *Aging protocols, chewing simulation, dental restorative materials, material durability, thermal cycling.*

Özet

Dental restoratif materyallerin uzun ömürlülüğü, başarılı klinik sonuçlar için kritik bir faktördür. Ağız içi ortam koşullarını taklit etmek amacıyla çeşitli in-vitro yaşlandırma protokolleri geliştirilmiştir. Bu protokoller arasında su içerisinde depolama, sodyum hipoklorit (NaOCl) ile temas, pH döngüleri, termal döngüler ve çiğneme simülasyonları yer almaktadır. Her bir yöntem; hidrolitik bozulma, kimyasal direnç, termal stabilite ve mekanik aşınma gibi dayanıklılık özelliklerine dair değerli bilgiler sunmaktadır. Ancak mevcut protokoller, ağız ortamının karmaşık ve dinamik yapısını tam anlamıyla yansıtmakta yetersiz kalmaktadır. Bu derleme, güncel yaşlandırma yöntemlerinin güçlü ve sınırlı yönlerini incelemekte ve klinik açıdan daha gerçekçi simülasyon sağlayacak kapsamlı protokollerin geliştirilmesi gerekliliğini vurgulamaktadır.

Anahtar Kelimeler: *Çiğneme simülasyonu, dental restoratif materyaller, malzeme dayanıklılığı, termal döngü, yaşlandırma protokolleri.*

OVERVIEW / GENEL BAKIŞ

The components of the oral environment can lead to the degradation of dental restorative materials, resulting in early failures. For dental restorations to have long-lasting durability, it is critical that the material's properties remain intact throughout their presence in the mouth.[1-4]

Clinically, restorations are routinely subjected to dynamic temperature fluctuations during breathing, eating, and drinking.[5] In vitro aging procedures for restorations aim to simulate oral conditions in the laboratory environment, reflecting the changes that intraoral conditions impose on materials.[6]

Aging of restorative materials can be simulated using mechanical, thermal, chemical, or combined methods.[7] The main factors influencing the severity of degradation in restorative materials are the temperature to which they are exposed and the aging duration.[1,7] Various aging methods that simulate the oral environment are utilized in studies to evaluate the behaviors of dental materials, such as water aging, NaOCl solution aging, pH cycling, food-simulating liquids, thermal cycling, mechanical occlusal loading (chewing simulator), and accelerated artificial aging.[8-12] This review aims to examine the aging methods applied in dentistry and analyze the studies conducted on this topic.

1. AGING PROCEDURES

1.1. Water Degradation

One of the most commonly used artificial aging techniques is water degradation. In this method, samples are kept in liquid at 37°C for a specific duration, which can range from several months to 4-5 years or even longer.[13] Many studies report significant reductions in bond strength after short storage periods.[14-16] The reduction in bonding efficacy is primarily assumed to result from the hydrolysis-induced degradation of interfacial components (primarily resin and/or collagen).[15] Distilled or deionized water is typically used as the storage medium in water degradation protocols.[16] Sodium azide, chloramine, or even antibiotics can be added to prevent bacterial growth during the storage period.[17,18] Artificial saliva solutions can also be used to more closely mimic clinical conditions, and some enzymes may be added to storage solutions for specific purposes.[16] For example, esterases produced by bacteria in vivo can catalyze the breakdown of resin components. Degradation depends on the rate of diffusion.[19,20]

1.2. Aging with NaOCl Solution

A more recent method for evaluating bond durability involves exposing the adhesive interface to an aqueous sodium hypochlorite (NaOCl) solution, which induces oxidation by creating superoxide radicals that break peptide chains.[21-23] Studies have shown that using a 10% NaOCl solution for a 5-hour storage period can dissolve the hybrid layer in both etch-and-rinse and self-etch adhesive systems, consequently reducing microtensile bond strength.[22-24] These findings suggest that the decrease in bond strength is related to the storage duration, as one hour of immersion in NaOCl solution is insufficient to completely dissolve the hybrid layer.[25] While this test is not recommended for evaluating the overall clinical performance of adhesive systems, if used in conjunction with prolonged water storage, it can provide broader insights into the stability of hybrid layer components.[22,24]

1.3. pH Cycling Aging

Oral pH varies depending on factors such as organic acids in plaque, bacterial metabolism, saliva, and dietary habits.[25] These factors, individually or collectively, can degrade composite resins or cause a loss of intermaterial bonds.[26] To evaluate the effect on composite resins, studies on pH cycling models have been conducted in recent years. In this test, designed by Featherstone and colleagues in 1986 to simulate clinical conditions, samples are immersed in an acid solution (pH 4.3 at 37°C for 6 hours) and then stored in artificial saliva at 37°C for 17 hours.[27] This cyclical effect can lead to liquid and bacterial ingress into the adhesive interface, adversely affecting bond strength. However, more studies are needed to better understand the chemical impact on resin materials and the resulting effect on bond strength.

1.4. Food-Simulating Liquids

Food-simulating liquids are used as a test method to evaluate the degradation mechanism of bonding and the mechanical properties of resin composites in vitro.[28] The liquids typically used to simulate foods include 10%, 50%, and 75% ethanol solutions, representing aqueous, acidic, and low-alcohol foods.[29] The reduction in fracture resistance after one year of storage in ethanol is primarily due to the softening and expansion of the resin matrix, leading to cracking within the resin.[30] Additionally, high ethanol diffusion may occur in adhesives containing HEMA.[29]

1.5. Thermal Cycling Aging

Thermal cycling is one of the most widely used in-vitro aging methods to simulate the thermal stresses that dental materials experience in the oral environment.[30] It replicates the effects of daily temperature fluctuations caused by the intake of hot and cold foods and beverages. These temperature changes induce repetitive contraction and expansion at the interface between the tooth and the restorative material due to differences in their coefficients of thermal expansion, potentially leading to marginal breakdown and reduced bond strength.[31,32]

In a typical thermal cycling protocol, specimens are alternately immersed in baths of cold (5°C) and hot (55°C) water, with a dwell time of 30 seconds in each bath and a transfer time of 5–10 seconds. The number of cycles varies among studies but generally ranges between 500 and 10,000 cycles, depending on the intended simulation period.[6,31]

This process mimics the intraoral thermal stress environment, where normal oral temperature averages around 35°C, and exposure to extreme temperatures during daily habits can range from

4.5°C to 55°C.[32-34] Thermal cycling has been shown to accelerate hydrolysis of the adhesive interface, especially when unprotected collagen is present, and to induce fatigue in polymerized resin structures.[34,35] Despite its widespread use, standardization across thermal cycling protocols is lacking, and variations in cycle count, temperature range, and dwell time limit cross-study comparisons.[6]

Therefore, refinement and standardization of thermal cycling parameters are crucial for ensuring the reliability and reproducibility of results in laboratory simulations of long-term material performance.

1.6. Mechanical Occlusal Loading Aging (Chewing Simulator)

Teeth are constantly subjected to stress due to functions such as chewing, swallowing, and parafunctional habits like bruxism.[37] The vertical force exerted by food between opposing teeth spreads across the entire occlusal surface. The resulting occlusal stress impacts the long-term survival rate of bonding and can also lead to mechanical degradation of the adhesive interface.[38] Chewing simulators are used to replicate in vitro stress similar to that occurring in vivo. These simulators can apply bidirectional force both horizontally and vertically. Dental materials are placed in plastic specimen holders, and predetermined horizontal and vertical movements are applied to simulate chewing.[37-39]

1.7. Accelerated Artificial Aging

Resin materials are highly affected by physical and chemical agents that can alter their optical properties. Stability tests conducted by increasing temperature are known as accelerated aging tests.[40] In these tests, products are exposed to temperatures above those typically encountered under normal conditions, reducing testing time and enabling faster results.[41] However, despite being accelerated, these tests are time-consuming and labor-intensive. To save time, UV aging tests are preferred.

The UV aging method is the most commonly used for accelerated aging tests. After accelerated aging, surface color changes have been reported in resin composites.[42-46] This method uses UV light, temperature, humid and dry air. Ultraviolet light, commonly known as UV light, is not visible to the naked eye and is primarily used in color studies and many other experiments. Xenon arc lamps are utilized in UV aging tests because they allow the application of realistic visible and UV light radiation.[43] This method is preferred for materials intended for exposure to natural light. Short-wavelength UV light contains more photon energy, leading to bond degradation in materials exposed to this light.[47] With increased temperature, the destructive effect is further enhanced. In particular, light-cured resin cements and resin composites containing light-sensitive aliphatic and aromatic tertiary amine groups are known to discolor more quickly when exposed to UV light due to the oxidation of these amine groups.[48-50]

DISCUSSION

The longevity and resilience of restorative materials in dentistry are heavily influenced by the complex interplay of physical, chemical, and thermal stresses within the oral cavity. Temperature fluctuations, pH variations, and the presence of biochemical enzymes collectively contribute to the gradual degradation of these materials, thereby underscoring the need for rigorous aging simulations to predict material performance. Various aging methodologies have been employed to capture these dynamics, each offering distinctive insights into specific stress factors impacting the clinical durability of restorations.

Oskoe et al. (2019) investigated the impact of esterase enzymes on dental composites, revealing that enzymatic degradation is a significant determinant in reducing the longevity of these materials.[1] Esterase, an enzyme naturally occurring in the oral environment, initiates hydrolytic degradation at the resin interface by breaking down the composite matrix structure. This process weakens the restorative material's structural integrity, compromising clinical success over time and emphasizing the need for materials that exhibit resistance to enzymatic activity in the oral cavity.[20]

Thermal changes are another critical factor in the degradation of dental materials. Di Giuseppe et al. (2018) demonstrated that restorative materials in the oral cavity undergo repetitive thermal expansion and contraction as a result of daily temperature changes, such as during eating and drinking.[5] These temperature-induced cycles introduce thermal stress into the resin matrix, progressively weakening its mechanical properties and reducing fracture resistance. Abo Elsoud et al. (2024) evaluated the durability of Bioflx, zirconia, and stainless steel crowns under thermomechanical aging protocols to simulate prolonged oral exposure.[32] Such findings highlight the importance of selecting materials that can endure the continuous thermal variations typical of the intraoral environment, a key criterion for achieving longevity in restorations.[33]

Chemical aging simulations, such as sodium hypochlorite (NaOCl) exposure, serve as essential tools for evaluating the stability of adhesive systems. Yamauti et al. (2003) demonstrated that NaOCl exposure leads to significant degradation within the hybrid layer, particularly in self-etch and etch-and-rinse adhesive systems, due to the breakdown of collagen at the adhesive interface.[22] Complementing this, De Munck et al. (2007) confirmed that NaOCl exposure compromises the structural integrity of HEMA-free adhesives, underscoring the importance of this method in assessing the long-term viability of adhesive systems.[13]

Topbaş et al. (2022) further explored the effects of various root canal irrigation protocols, including NaOCl, on bond strength between dentin and ceramic posts.[11] Their findings indicate that NaOCl irrigation followed by mechanical aging significantly reduces micro pushout bond strength (mPBS), especially in the apical zone of the root canal. This study expands the scope of chemical aging, showing its relevance not only in adhesive systems but also in endodontic bonding, where irrigation solutions play a crucial role in bond longevity.

The pH cycling method, which simulates alternating acidic and neutral exposure, provides a valuable framework for understanding the effects of pH fluctuations due to diet or bacterial metabolism on the bonding strength of restorative materials. Featherstone et al. (1986) demonstrated that acidic conditions weaken bonding strength by facilitating microleakage at the adhesive interface, potentially leading to premature failure.[27] Bagheri et al. (2005) extended this by examining food-simulating solutions (FSS), finding that ethanol, a common component in various foods and beverages, compromises composite mechanical properties by penetrating the resin matrix and disrupting internal bonds.[28] This results in surface softening, expansion, and eventual microcracking, highlighting the necessity of dietary considerations in restorative material selection.

Drubi-Filho et al. (2012) examined accelerated artificial aging (AAA) using UV light and thermal exposure, observing significant color instability and structural degradation in composites with high water absorption.[42] Monticelli et al. (2007) found that UV light exposure causes rapid chemical degradation in resin composites, leading to surface cracking and discoloration.[25] Similarly, Narde et al. (2024) demonstrated that PMMA is more susceptible to color changes and surface degradation under thermal aging compared to indirect composites.[9] These findings highlight the importance of incorporating environmental factors unique to the oral cavity in aging protocols, especially for anterior restorations, where esthetic stability is paramount.

In the realm of digital advancements, Alageel et al. (2022) reported that 3D-printed and milled interim restorations exhibit higher flexural strength and smoother surfaces post-aging compared to conventionally fabricated counterparts.[39] This suggests that CAD-CAM and 3D printing techniques may yield more durable interim restoration options, offering superior structural stability and resilience under accelerated aging conditions.

Mechanical aging methods, such as thermal cycling and chewing simulations, are fundamental to evaluating material resilience under conditions that mimic occlusal forces and temperature fluctuations. Thermal cycling tests replicate the stress caused by daily hot and cold cycles, which introduces expansion-contraction cycles that degrade adhesive integrity. El Araby and Talic (2007) demonstrated that such cycles accelerate collagen hydrolysis within the hybrid layer, leading to resin dissolution and bond degradation.[31] Chewing simulators, which replicate the occlusal forces and multidirectional stresses of mastication, are also crucial. Vilde et al. (2022) confirmed that chewing simulators reliably simulate in vivo conditions, providing a robust assessment of wear resistance and mechanical stability under repeated occlusal loading.[40]

Each of these aging methodologies—enzymatic, chemical, thermal, dietary, and mechanical—provides invaluable insights into different aspects of material degradation under simulated oral conditions. However, limitations remain, underscoring the need for more advanced and comprehensive aging protocols. Future research should prioritize developing methodologies that more accurately reflect the complexity of intraoral conditions, including dynamic pH changes, mechanical loads, and enzymatic interactions. Such advancements will support the creation of next-generation dental materials that offer enhanced durability, esthetic stability, and clinical efficacy, ultimately leading to improved patient outcomes.

SUMMARY / SONUÇ

The durability of restorative materials in the oral environment is crucial for long-term clinical success. Aging methods that assess wear caused by temperature changes, pH fluctuations, enzymatic activity, and mechanical stress reveal the vulnerabilities of these materials. Techniques such as chewing simulators and accelerated aging tests offer valuable insights into the clinical performance of these materials, although limitations in current methods must be considered. In the future, the development of tests that better simulate clinical conditions will pave the way for more resilient and successful restorations.

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