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Valuation the Oil leaching and the Degree of Conversion of Addition of Miswak (Salvadora Persica) Essential Oil to Orthodontic Adhesive

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ABSTRACT: This study inspects the Oil leaching and the Degree of Conversion of Addition of Miswak (Salvadora Persica) Essential Oil to Orthodontic Adhesive. Miswak oil was prepared from the Miswak root by the green method, and was added to the Heliosit adhesive at 1%,3% and 5% respectively. The degree of conversion was measured by Fourier Transform Infrared Spectroscopy, and the amount of leached oil was estimated by Spectrophotometer. The degree of conversion data was analyzed by Friedman test where $p \leq 0.05$. The degree of conversion was not affected by the oil added to the adhesive. While the leached oil of the modified adhesive started from the first hour of curing and extended for last day of the study (one month). According to this study the addition of Salvadora Persica oil did not affect the degree of conversion of the modified adhesive. Also, the amount of oil leached from the modified adhesive began on the first day of polymerization and continued for up to one month.

Keywords: Degree of Conversion, Orthodontic adhesive, Miswak oil, Oil leaching

1. INTRODUCTION

In recent years, the world has increasingly embraced the power of herbal remedies to tackle diverse health issues. This surge in recognition is fueled by the remarkable effectiveness of herbal therapies and their gentle, side-effect-free nature [1]. The World Health Organization (WHO) reports that a substantial portion of the global population, especially in less developed regions, depends on traditional herbal treatments and natural medicines to meet their healthcare needs [1, 2]. Consequently, the WHO has championed the integration of medicinal herbs into the healthcare systems of developing countries to boost overall healthcare effectiveness. Among these herbal marvels is Salvadora Persica, often hailed as the 'miracle twig,' which has garnered widespread support for its remarkable benefits [3]. Salvadora Persica, more commonly known as Miswak, is a proud member of the Salvadoracea plant family [4], It thrives mainly in the arid and subtropical landscapes of the Middle East, the Indian subcontinent, and Africa [5], The branches, fresh leaves, and roots of this tree have long been treasured in herbal remedies, traditionally used to treat conditions like coughs, asthma, scurvy, and to maintain oral hygiene [6]. Historical records reveal that its use dates back to both pre-Islamic and Islamic eras. During these times, it was introduced in Arabic and Islamic societies as an early form of toothbrush, aiding in dental cleaning and promoting oral hygiene [7], The dental benefits of Salvadora Persica stem from its mechanical cleaning action during brushing and its rich bioactive components. Notably, tannins in the plant inhibit the glucosyltransferase enzyme, reducing plaque formation and preventing periodontal diseases. Additionally, resins offer protection against tooth decay [8]. The recent wave of interest in natural and herbal products, such as Salvadora Persica, for periodontal care is particularly promising. It offers potential benefits, especially for economically disadvantaged communities around the world. Salvadora Persica

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stands as a deeply rooted herbal therapy, intertwined with culture and religion. Its rich history spans diverse regions worldwide, where it has been used to treat a variety of health conditions [9, 10], SP, a plant native to the arid landscapes of the Middle East and Africa and widely cultivated across the Indian Peninsula, has gained popularity as a common herbal remedy throughout various historical epochs [7].

Resin composites primarily consist of an organic matrix, inorganic fillers, and an initiation system. The organic matrix usually includes bisphenol-A glycol dimethacrylate (Bis-GMA), urethane dimethacrylate (UDMA), and triethylene glycol dimethacrylate (TEGDMA). During polymerization, the initiation system facilitates a crosslinking reaction that breaks the carbon-carbon double bonds (C=C) within the dimethacrylate monomers, converting them into carbon-carbon single bonds (C-C). This reaction leads to the formation of a polymer network. On a microscopic level, the intermolecular forces between monomers transition from van der Waals interactions to covalent bonds, causing a reduction in intermolecular distances. As a result, the resin composite experiences a decrease in overall volume on a macroscopic scale [11]. After the polymerization of monomers, a small amount of residual monomers often remains within the cured product. The degree of conversion (DC) is used as an indicator of resin composite polymerization, representing the percentage of polymerizable double bonds that have been converted into single bonds. In resin composites, the DC typically ranges from 52% to 75%.[12]. The degree of conversion (DC) affects the mechanical and physical properties of the material. A lower DC results in a reduced crosslink density within the polymer, which leads to weaker mechanical properties, increased susceptibility to discoloration and degradation, and consequently, diminished wear resistance and color stability. [13]. Additionally, a lower DC compromises the biosafety of materials due to the increased release of unpolymerized monomers. Therefore, an ideal resin composite should exhibit minimal polymerization shrinkage while maintaining a high DC. Among the various techniques for assessing DC, Fourier transform infrared spectroscopy (FTIR) is the most commonly used, offering both qualitative and quantitative insights into the material's degree of conversion. [14]. Polymer science has emerged as a leading field of study due to its broad applications in modern materials engineering, especially in enhancing structural and functional properties in clinical and biomedical contexts. Many polymers exhibit ideal characteristics that can be further optimized for bioactivity, cytocompatibility, and antibacterial properties through various chemical modifications. [15]. Polymers are extensively used in orthodontic applications due to their excellent mechanical properties and aesthetic appeal. These orthodontic polymers are classified into several categories, including adhesives, brackets, elastomeric modules, and chains. [16], A wide range of orthodontic polymers is available on the commercial market, including polycarbonate, polyurethane, polyethylene, polyamides, polymethyl methacrylate, and others [17, 18]. In recent years, attention has focused on the potential release of Bisphenol-A (BPA) from polycarbonate brackets and monomers, especially in orthodontic composites containing bisphenol A-glycidyl methacrylate (bis-DMA)[19]. BPA leaching can occur due to incomplete polymerization of monomers, which remain as impurities after resin synthesis, as well as the degradation of polycarbonate materials [20]. Despite modifications to monomer structures and molecular interactions, several clinical measures can help mitigate the release of Bisphenol-A (BPA) from orthodontic materials. Recommendations include positioning the light-curing tip of the lamp close to the composite during curing to ensure thorough polymerization. Employing indirect light-curing techniques, such as directing light onto the edges of the bracket rather than through it directly, can also help reduce BPA exposure. Additionally, polishing the composite with a pumice stone after bonding may further minimize BPA release. Patients should be advised to rinse their mouths with mouthwash or water within the first hour after composite placement to decrease exposure [21]. To date, no studies have evaluated the effect of adding SP essential oil to orthodontic composites used for bonding brackets to teeth during orthodontic treatment. This study aimed to assess the amount of oil leached from an orthodontic adhesive modified with Miswak oil and to calculate its degree of conversion.

2. MARERIALS AND METHODS

The Study Ethics Team of the College of Dentistry at the University of Mosul granted ethical clearance (UOM Dent/H.DM. 51/22), the criteria for teeth selection included ensuring that the teeth were free from hypoplastic areas, caries, attrition, cracks, gross irregularities, and restorations. Additionally, the selected teeth had not undergone orthodontic or endodontic therapy and had no history of prior treatment with chemical substances such as alcohol, formalin, or H_2O_2 [22]. The dentists who collected the samples verified this information. The labial surfaces of the teeth were examined at x10 magnification using a microscope connected to a laptop to confirm their surface integrity. The samples were then stored in distilled water at room temperature (22°C ± 2) for up to one month [23, 24]. The sample size was collected according to previously published articles as ten sample per each group to get 80%power of the study [23-25]. Thus, the study samples consisted of 40 human premolar teeth (first and second, upper and lower premolars) extracted for orthodontic purposes from patients aged 16-25 years. These teeth were obtained from Dental Health Centers and various private dental clinics in Mosul city.

2.1 Study Design

The experimental design for SP essential oil leaching involved preparing 3 discs with modified adhesives as follows:

1. Group One (0%): Three discs of the orthodontic adhesives were prepared and light-cured without the addition of any additives.

2. Group two (3%): Three discs of the orthodontic adhesives were prepared and light-cured with the addition of 3% SP essential oil.

Each group was immersed in distilled water within dark, sterile containers for specified time intervals (1 hour, 24 hours, 1 week, 1 month). All samples were maintained in a laboratory incubator at 37°C to replicate the temperature of the mouth.

The degree of conversion (DC) was evaluated by preparing 5 discs of the modified adhesives as follows:

1.Group One (0%): Five discs of orthodontic adhesive were prepared and light-cured without any additives.

2. Group Two (1%): Five discs of orthodontic adhesive were prepared and light-cured with the addition of 1% SP essential oil.

3. Group Three (3%): Five discs of orthodontic adhesive were prepared and light-cured with the addition of 3% SP essential oil.

4. Group Four (5%): Five discs of orthodontic adhesive were prepared and light-cured with the addition of 5% SP essential oil.

A fresh Salvadora persica root was sourced from a male Arak tree in the Ha'il region of Saudi Arabia. To prevent bacterial contamination, the roots were cleaned with alcohol before use [26]. The root was then cut into small pieces and ground into a powder using a clean home grinder (SUS304, China). This powder was placed in the chamber of a Soxhlet apparatus, a specialized device for extracting essential oil from plants, as illustrated in Figure 1.

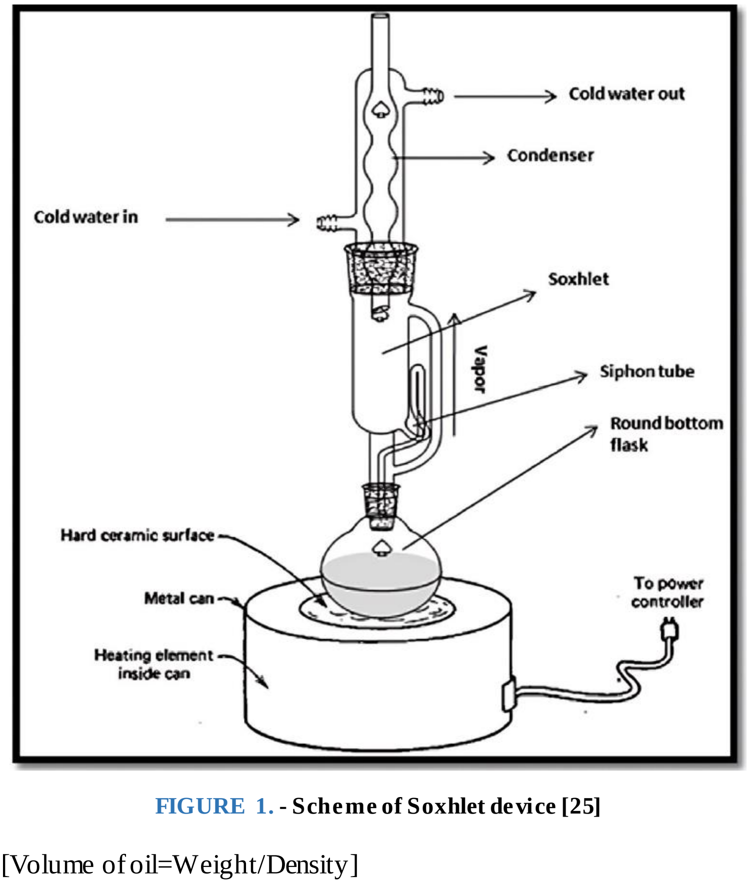

FIGURE 1. - Scheme of Soxhlet device [25]

Using the formula [Volume of oil=Weight/Density]

and a density of 0.9246 mg/µL, the volume of essential oil required was calculated ≈10.82 µL

Approximately 10.82 µL of SP essential oil was added to 1000 mg of orthodontic adhesive to achieve a 1% concentration of modified adhesive with SP essential oil.

Based on this calculation, the groups were prepared as follows:

1. Control group (0%): No oil was added to the adhesive.

2. 1% group: 10.82 µL of SP essential oil was added to 1000 mg of Heliosit adhesive.
3. 3% group: 32.45 µL of SP essential oil was added to 1000 mg of Heliosit adhesive.
4. 5% group: 54.09 µL of SP essential oil was added to 1000 mg of Heliosit adhesive.

Each mixture was then spread evenly on a sterile, clean glass slab to minimize porosity [26]. Specimens consisting of three discs each, categorized as the control group and the 3% SP essential oil group, were fabricated using plastic molds. The discs were standardized to a diameter of 3 mm and a thickness of 2 mm [27], After filling the molds with adhesive, a celluloid matrix strip was applied. The specimens were then subjected to complete light-curing using a Woodpecker device (Jiangsu, China) for a total of 40 seconds (20 seconds for both the top and bottom). Following this, the cured specimens were carefully removed from the molds. The assessment of light absorption leaching from the discs was carried out using a spectrophotometer. To evaluate the leaching of oil from the modified adhesive, an ultraviolet (UV) visible spectrophotometer was utilized. Samples were placed in the spectrophotometer, which was set to scan wavelengths between 200 and 500 nanometers (nm). The absorbance of the oil was specifically monitored at 280 nm, a wavelength where SP oil is known to exhibit absorbance. This wavelength is commonly associated with the absorbance of aromatic amino acids (such as tryptophan and tyrosine) and phenolic compounds, which are frequently found in plant-derived oils and extracts [28], as shown in figure (2).

FIGURE 2. - Fresh glass cups containing (0%, 3%, SP essential oil modified adhesive discs) rinsed in water for oil leaching tests

An ultraviolet-visible spectrophotometer (Shimadzu UV-1800, Japan) was used for the analysis. The initial analysis was performed on distilled water alone, and subsequent measurements were taken at one-hour intervals and beyond. These measurements were used to generate charts that allowed for the determinant. To calculate the degree of conversion (DC) of the modified orthodontic resin, five specimen discs were prepared for each group (0%, 1%, 3%, 5%). Each disc measured 5 mm in diameter and 1 mm in thickness and was produced using a clear mold. A 0.5 mm-thick celluloid strip was placed on a glass surface, and the resin material was overfilled into the mold, which was positioned on a yellow background with 70% reflectivity. The surface of the resin was then covered with another glass slide and celluloid strip. Light activation of all specimens was performed using a fully charged LED curing unit (Woodpecker, Jiangsu, China) in standard mode, with a light intensity of 1700 mW/cm². The unit was positioned at a distance of 0 mm from the specimens, and each specimen was cured for 20 seconds on the top surface and 20 seconds on the bottom surface. Following light curing, the samples were stored in distilled water at room temperature (22°C ± 2) for 24 hours to accommodate any post-curing effects. After this period, the specimens were carefully removed from the molds [29]. Infrared spectral bands were measured using an Alpha II Platinum spectrophotometer (Bruker Optic GmbH, Ettlingen, Germany) with a resolution of 2 cm^{-1}, covering the wavelength range of 400-1800 cm^{-1}, and connected to a laptop (see Figure 3). The measurements included uncured orthodontic adhesive, cured orthodontic resin alone, cured orthodontic adhesive modified with 1% SP essential oil, cured orthodontic adhesive modified with 3% SP essential oil, cured orthodontic adhesive modified with 5% SP essential oil, and SP essential oil alone (50 ml). [30, 31].

FIGURE 3. - Fourier-transform infrared spectrophotometer (Alpha II, Platinum, Bruker Optic GmbH, Ettlingen, Germany)

The degree of conversion (DC) of the polymerized samples was determined using Attenuated Total Reflectance (ATR) Fourier Transform Infrared Spectroscopy (FTIR) with the ATR/FTIR Alpha II Platinum (Bruker Optic, Germany), conducted in absorbance mode. While there are various methods for calculating DC%, FTIR is a precise and straightforward technique that measures the interaction of light with the material to assess the degree of polymerization of monomer molecules. In conventional methacrylate-based compositions, the aliphatic C=C bonds (which have an analytical peak at 1637 cm^{-1}) are converted to C-C bonds during polymerization. The aromatic C=C bonds (at 1610 cm^{-1}), which are not affected by the polymerization reaction, serve as an internal reference peak. This reference allows for accurate measurement of the degree of conversion without requiring precise knowledge of the component concentrations. [29], The degree of conversion (DC%) was calculated using the following equation:

[% DC = 100 × 1 – (AP (C=C) × AM (C..C)/ AM (C=C) × AP (C..C))]

where A (C=C) represents the absorbance of the aromatic C=C bonds (1610 cm^{-1}) and A(C..C) represents the absorbance of the aliphatic C=C bonds (1637 cm^{-1}) after polymerization.

3. Results

The study detected leaching of essential oil from the 3% SP oil-modified adhesive at multiple time points: 1 hour, 24 hours, one week, and one month. This leaching was observed at a wavenumber of 280 nm, corresponding to the absorbance area of SP oil in the UV-Visible Spectrophotometer (Shimadzu, UV-1800, Japan). The findings highlight the progressive nature of essential oil leaching over time, with the amount of leaching at one month exceeding that at one week, which in turn was greater than at 24 hours and one hour. In contrast, the control group exhibited no oil leaching, as it contained no SP oil additives, as illustrated in Figure (4).

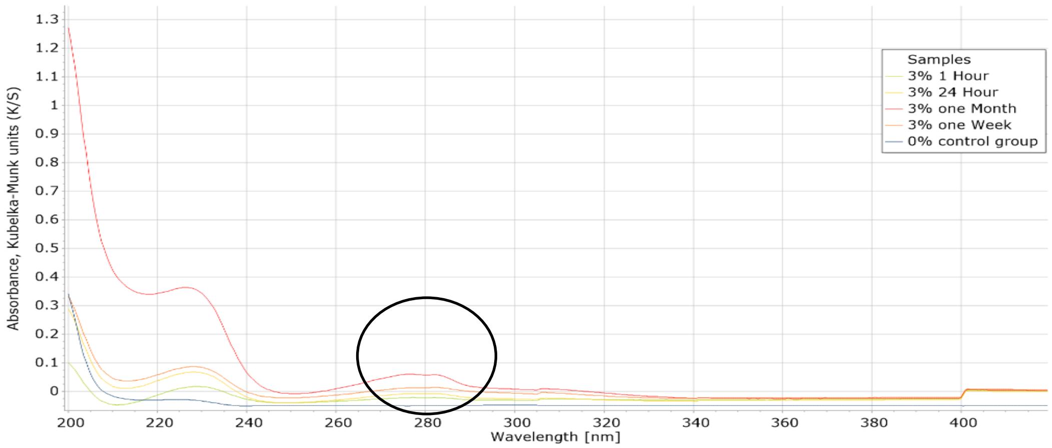

FIGURE 4. - Schematic chart shows the curves of UV- Visible Spectrophotometer (Shimadzu, UV-1800, Japan) in absorbance and wavenumber axis for 3% SPO modified adhesive

Table (1) presents descriptive statistics for the degree of conversion (DC), including mean values, standard deviations, and maximum and minimum values across the study groups. The analysis revealed that the control group, with 0% SPO-modified adhesive, exhibited the highest mean DC value of 47.79%. This was followed by the 1% SPO-modified adhesive, which had a mean DC value of 41.40%. The 3% SPO-modified adhesive ranked third with a mean DC value of 35.52%, while the 5% SPO-modified adhesive demonstrated the lowest mean DC value of 27.67%.

Table 1. - Descriptive statistics for degree of conversion

Samples		Minimum	Maximum	Mean	Std. Deviation
0% Control group	5	61.19	41.79	47.46	7.855
1% SPO*	5	58.21	31.34	41.40	10.594
3% SPO	5	50.75	14.93	35.52	14.017
5% SPO	5	32.84	17.91	27.67	5.858

*SPO represents SP oil modified adhesive

Figure (5) provides a clear depiction of the FTIR/ATR waves, representing the degree of conversion (DC) of modified adhesives at various concentrations. The concentration range of interest lies between 1607 and 1638 cm^{-1}, which indicates the DC of the adhesive monomer. As illustrated in Figure 4.9, the 5% SPO-modified adhesive exhibits the highest absorbance level within this range, followed by the 3% and 1% SPO-modified adhesives. The control group shows the lowest absorbance level.

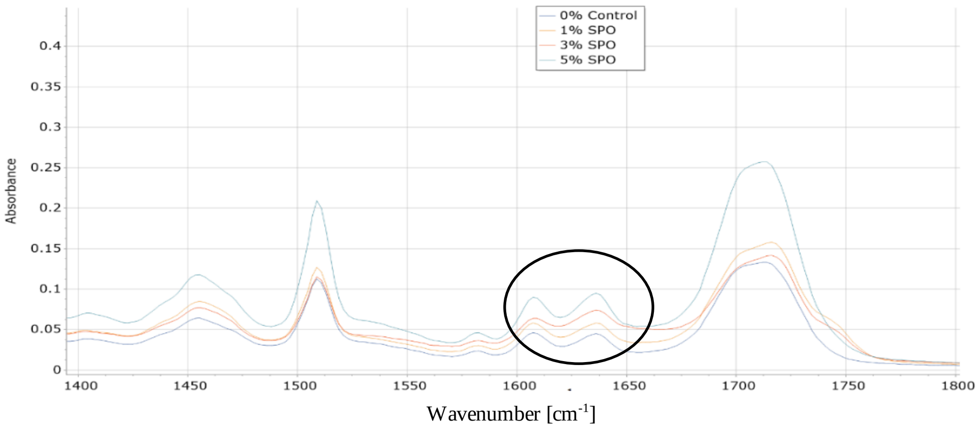

FIGURE 5. - Schematic view of FTIR/ATR that shows degree of conversion of 0%, 1%, 3%, 5% SP oil modified adhesive

Given that there were three repeated samples for each of the five groups, a Friedman analysis was conducted. The results indicated a p-value of less than 0.05, suggesting that there were no differences between the groups. These findings are presented in Table (2).

Table 2. - Friedman analysis for degree of conversion

Friedman Test	
Groups	5
Test Statistics	7.800
DF*	3
Asymp. Sig.	0.052

*DF represents degree of freedom. $p \leq 0.05$

4. Discussion

Throughout human history, herbs and their essential oils have been used for various purposes, including masking odors, enhancing social appeal, and improving culinary products, as well as in perfumery and cosmetics. Their medicinal benefits are also well-documented, encompassing a range of biological properties such as larvicidal, analgesic, anti-inflammatory, antioxidant, fungicidal, and antitumor activities, among others. To test oil leaching, three discs from each group—the control (0%) and the 3% SP oil-modified adhesive—were selected at various time intervals: one hour, 24 hours, one week, and one month. An absorption peak at 280 nm appeared typically indicating the presence of aromatic amino acids like tryptophan and tyrosine, or other aromatic compounds. Therefore, when analyzing the UV-Vis spectrum of SP essential oil and observing an absorption peak around 280 nm, it is likely due to the presence of these aromatic compounds [32]. In the UV-Vis spectral analysis of SP essential oil (SPO), a notable increase in the absorption curve around 280 nm was observed, with the intensity becoming more pronounced over time. Specifically, after one month, there was higher leaching of SPO compared to the shorter time frames of one hour, 24 hours, and one week. This suggests that the concentration of phenolic compounds and other aromatic constituents increases with prolonged exposure, indicating progressive extraction or leaching of these compounds from the plant material over time. The observed increase in SPO leaching over time can be attributed to the behavior of the adhesive fillers within the polymer matrix. As time progresses, these fillers absorb water and swell through a process known as imbibition. This swelling facilitates the sustained release of the embedded oil, resulting in a higher concentration of leached oil at the one-month mark compared to shorter durations such as one hour, 24 hours, and one week. This prolonged release mechanism highlights the dynamic interaction between the adhesive fillers and the aqueous environment, leading to enhanced oil leaching over time [33].

The study of the absorption curve at 230 nm revealed the release of the monomer, as this wavelength corresponds to the monomer's absorption peak. The monomer release was monitored at various time intervals (1 hour, 24 hours, 1 week, and 1 month) to assess the impact of oil addition on the leaching process. The incorporation of SP essential oil affected the monomer's leaching. The absorption curves demonstrated that the presence of the oil altered both the rate and extent of monomer release. At each time interval, samples containing the oil exhibited distinct absorption characteristics compared to those without the oil, suggesting that the oil either facilitated or modulated the leaching process [34].

Investigating the degree of conversion (DC) of orthodontic resins in relation to material viscosity is crucial for identifying potential variations among different resin composite brands regarding their leaching tendencies of unpolymerized constituents. Such analyses are important for assessing the potential cytotoxicity to oral cell populations. Therefore, understanding the relationship between DC and material viscosity in orthodontic resin systems is essential for evaluating their safety and effectiveness [35, 36].

The degree of conversion (DC) for the control group in our study was consistent with the range reported in previous studies [36, 37]. A degree of conversion (DC) of 48.63 was reported in prior research, which closely aligns with our DC of 47.46, as both studies used the same protocol for measuring the DC of the orthodontic adhesive. To assess the DC, the Friedman test was employed due to the non-parametric nature of the repeated measurements, whereas parametric data would typically be analyzed using ANOVA. The results indicated that the degree of conversion (DC) decreased with increasing amounts of SP essential oil (SPO) added to the adhesive. This decline is attributed to the incorporation of the oil into the spaces between the adhesive fillers [29] For further researches we suggest an exploration of leaching durability through long-term (six months) studies to assess the adhesive's ability to maintain its properties and performance over an extended period, providing crucial data for predicting its clinical longevity and stability.

A key limitation of this study is its reliance on only one type of orthodontic composite. Furthermore, the study did not incorporate pH cycling or thermocycling, which might influence the results.

5. Conclusions

The addition of SP oil did not affect the degree of conversion of the modified adhesive. According to this study, the amount of oil leached from the modified adhesive began on the first day of polymerization and continued for up to one month.

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CONFLICTS OF INTEREST

The authors declare no conflict of interest

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