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Versiegelung 3D gedruckter zahnärztlicher Restaurationen mittels Malfarbe

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Abkürzungsverzeichnis

ISO – International Organization for Standardization

T0 – Tag 0

T14 – Tag 14

IP – Individuelle Prophylaxe

PP – Professionelle Prophylaxe

Publikationsliste

Lask M, Stawarczyk B, Reymus M, Meinen J, Mayinger F. Impact of varnishing, coating, and polishing on the chemical and mechanical properties of a 3D printed resin and two veneering composite resins. J Prosthet Dent. 2024 Aug;132(2):466.e1-466.e9. doi: 10.1016/j.prosdent.2024.05.006. Epub 2024 May 25. PMID: 38797579.

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Der eigene Beitrag zu der Promotion sowie zu den beiden Publikationen „Impact of varnishing, coating, and polishing on the chemical and mechanical properties of a 3D printed resin and two veneering composite resins“ und “Impact of Glazing, Coating, and Polishing on the Color Stability and Surface Properties of a 3D Printed Resin and Two Veneering Composite Resins“ kann der folgenden Tabelle entnommen werden.

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1. Einleitung

Fortschritte in der 3D-Drucktechnologie und die Entwicklung hochleistungsfähiger Harzmaterialien haben die Herstellung von permanentem festsitzendem Zahnersatz ermöglicht [1, 2]. Diese additiv gefertigten Kunststoffe bieten eine kost- und zeiteffiziente Alternative zu konventionellen Restaurationsmethoden, einschließlich metall- und keramikbasierter Versorgungen. Trotz dieser Vorteile sind die mechanischen, chemischen und optischen Eigenschaften 3D-gedruckter Materialien für den langfristigen klinischen Einsatz noch nicht vollständig optimiert [3-7]. Die mechanischen Eigenschaften eines Materials, wie z.B. Biegefestigkeit, Abrasionsbeständigkeit und Härte, sind entscheidend für die Langlebigkeit definitiver festsitzender prothetischer Versorgungen [8-10]. Ebenso spielen chemische Eigenschaften wie die Biokompatibilität sowie oberflächenbezogene Parameter, darunter Rauigkeit und freie Oberflächenenergie, eine wesentliche Rolle für den langfristigen Erfolg eines zahnärztlichen Materials [11-14]. Eine homogene, glatte Oberfläche mit einer Rauigkeit von unter $0,2\ \mu\text{m}$ ist erforderlich, um die Anlagerung von Plaque – und damit die Ursache für Gingivitis, Parodontitis, und Candidiasis – weitgehendst minimieren zu können und den mechanischen Verschleiß des Restaurationsmaterials sowie des Antagonisten reduzieren zu können [15-17].

Eine der größten Herausforderungen 3D-gedruckter Harze ist ihre eingeschränkte Farbstabilität. Verfärbungen treten häufig auf und sind einer der Hauptgründe für den Austausch polymerbasierter Restaurationen [5, 18, 19]. Farbveränderungen entstehen sowohl durch extrinsische Faktoren [20-22] wie Farbstoffe in Nahrungsmitteln (z. B. Rotwein, Kurkuma), als auch durch intrinsische Prozesse innerhalb des Materials [20, 23, 24]. Oberflächenrauigkeit spielt dabei eine wesentliche Rolle, da eine raue Oberfläche eine größere Kontaktfläche für Farbpigmente bietet und damit deren Adhäsion begünstigt [20, 25, 26]. Zusätzlich beeinflusst die Oberflächenenergie die Benetzbarkeit: Ein niedriger Kontaktwinkel führt zu einer verstärkten Farbaufnahme und erhöhtem Verfärbungsrisiko [20, 27, 28].

Um die mechanischen und optischen Eigenschaften von 3D-gedruckten Harzen zu verbessern, und diese langfristig klinisch am Patienten einsetzen zu können, können

verschiedene Oberflächenbehandlungen zum Einsatz kommen. Zu den etablierten Methoden gehören Polieren, Lackieren und Beschichten („Coating“). Eine Politur der Oberfläche kann als bewährte Technik zur Reduktion der Oberflächenrauigkeit angesehen werden und kann sowohl die ästhetische Erscheinung als auch die mechanischen Eigenschaften der Restauration verbessern [27, 29-31]. Alternativ kann auch das Auftragen einer polymerisierbaren, niedrigviskosen Glasur durch Lackieren oder Beschichten eine versiegelte, homogene und glatte Oberfläche erzeugen sowie die Farbstabilität dentaler Materialien erhöhen [29, 30, 32-34]. Während Politurverfahren in der Literatur bereits ausreichend untersucht wurden, gibt es bisher nur wenige Studien, die die Effekte von Lackieren oder Beschichten auf die Oberfläche von 3D-gedruckten Restaurationen systematisch analysieren [15, 34-36].

Neben einer sachgerechten Nachbearbeitung der Materialoberfläche können auch Verfärbungen durch individuelle oder professionelle Prophylaxe verhindert bzw. rückgängig gemacht werden [20, 30]. Diese Reinigungsmöglichkeiten sind hinsichtlich ihres Einflusses auf 3D-gedruckte Harze ebenfalls noch nicht hinreichend untersucht worden.

2. Zielsetzung

In den zwei dieser Rahmenschrift zugrundeliegenden international veröffentlichten Publikationen wurde der Einfluss verschiedener Oberflächenbehandlungen (Polieren, Lackieren, Beschichten) auf ein 3D-gedrucktes Harz (VarseoSmile Crown^{Plus}, BEGO) und zwei Verblendkomposite (GRADIA PLUS, GC EUROPE NV; VITA VM LC flow, VITA Zahnfabrik) hinsichtlich mechanischer, chemischer und optischer Materialeigenschaften untersucht. Als Versuchsparameter wurden die Martens-Parameter, die biaxiale Biegefestigkeit, die Abrasionsbeständigkeit (Drei-Körper-Verschleiß), die Umsetzungsrate, die Oberflächenrauigkeit, die freie Oberflächenenergie und die Verfärbungsrate nach Lagerung in verschiedenen Farbmedien (Rotwein, Kurkuma, Kresse, Wasser) bestimmt und anschließend statistisch ausgewertet (IBM Statistics SPSS 29.0, IBM, Armonk, USA). Zudem wurde untersucht, inwiefern individuelle und professionelle Prophylaxemaßnahmen mögliche Verfärbungen wieder rückgängig machen können. Hier wurde im Rahmen der 2. Untersuchung eine im Rahmen dieser Dissertation selbstentwickelte und - konstruierte Zahnbürstenapparatur verwendet, die ein standardisiertes Vorgehen zur Durchführung der Zahnreinigung ermöglichte.

3. Material und Methoden

Als Materialien wurde das 3D-Druck Harz VarseoSmile Crown^{plus} (BEGO), sowie die Verblendkomposite GRADIA PLUS (GC EUROPE NV) und VITA VM LC flow (VITA Zahnfabrik) verwendet. Während das 3D-gedruckte Material im additiven Verfahren mittels 3D-Drucker hergestellt wurde, erfolgte die Herstellung der Prüfkörper aus Verblendkompositen durch schichtweises Einbringen des Materials in Silikonformen nach dem Inkrementverfahren. Die anschließende Aushärtung wurde jeweils gemäß den Vorgaben der Hersteller durch Lichtpolymerisation durchgeführt. Nach der Fertigung und sachgerechten Nachbearbeitung der 3D-gedruckten Prüfkörper wurden verschiedene Verfahren zur Oberflächenbehandlung angewendet: die Politur mit Ziegenhaarbürstchen in Kombination mit einer Polierpaste (Abraso Starglanz, bredent), die Politur mittels Silikonpolierern (Sirius ceramics), das Auftragen eines Lacksystems (OPTIGLAZE color, GC EUROPE NV bzw. VITA AKZENT LC, VITA Zahnfabrik), sowie die Anwendung eines Coating-Verfahrens. Letzteres beinhaltet das Auftragen desselben niedrigviskösen Harzes (VarseoSmile Crown^{plus}) auf die Oberfläche der Prüfkörper, gefolgt von einer erneuten Aushärtung gemäß Herstellerangaben. Für die Verblendkomposite wurde ausschließlich eine Politur mittels Ziegenhaarbürstchen und Silikonpolierern als Oberflächenbehandlung durchgeführt. Prüfkörper, die keiner Oberflächenbehandlung unterzogen wurden, dienten als Kontrollgruppen. Nach Abschluss der Herstellung sowie der jeweiligen Oberflächenbearbeitung wurden alle Prüfkörper für eine Dauer von sieben Tagen in destilliertem Wasser bei einer konstanten Temperatur von 37 °C in einem Inkubator (HeraCell) gelagert.

Untersuchung I

Im Rahmen der ersten Untersuchungsreihe wurden insgesamt 288 Prüfkörper hergestellt. Davon dienten 144 scheibenförmige Prüfkörper ($\varnothing 12 \times 1,2 \pm 0,2$ mm) der Bestimmung der chemischen Umsatzrate, der Oberflächenrauigkeit, der Martens-Parameter, sowie der biaxialen Biegefestigkeit. Weitere 144 rechteckige Prüfkörper ($12 \times 10 \times 4$ mm) wurden für die Untersuchung der Abrasionsbeständigkeit mittels ACTA-Maschine (ACTA 3; SD Mechatronik) verwendet. Zur Charakterisierung der Prüfkörper wurden verschiedene Materialeigenschaften analysiert: Die chemische Umsatzrate wurde mithilfe der Raman-Spektroskopie (inVia Qontor; Renishaw) bestimmt, während die Oberflächenrauigkeit mit einem Kontaktprofilometer (MarSurf M400; Mahr) erfasst

wurde. Die Martens-Parameter, bestehend aus der Martenshärte und dem elastischen Eindringmodul, konnten durch instrumentierte Eindringprüfung mit einem Vickers-Diamanten (ZHU 0.2; ZwickRoell) ermittelt werden. Abschließend erfolgte die Bestimmung der biaxialen Biegefestigkeit gemäß DIN EN ISO 6872:2019 unter Verwendung einer Universalprüfmaschine (Z10; ZwickRoell). Zur Bestimmung der Abrasionsbeständigkeit im Rahmen eines Drei-Körper-Verschleißtests wurde ein Prüfkörperad zusammen mit einem Antagonistenrad in einer abrasiven Hirsemischung in der ACTA-Maschine über 200.000 Zyklen getestet. Anschließend erfolgte die Visualisierung und Auswertung des Volumenverlusts mittels Laserscanner (LAS-20; SD Mechatronik).

Bei der statistischen Auswertung wurde die Abweichung von der Normalverteilung der Daten mit dem Kolmogorov-Smirnov-Test überprüft; bei fehlender Normalverteilung kamen nichtparametrische Verfahren wie der Mann-Whitney-U-Test, der Kruskal-Wallis-Test sowie der Spearman Korrelationstest zur Anwendung ($\alpha=0.05$).

Untersuchung II

Im zweiten Teil der Untersuchung wurden insgesamt 576 scheibenförmige Prüfkörper ($\emptyset 16 \times 1,2 \pm 0,2$ mm) hergestellt. Diese setzten sich zu gleichen Teilen aus dem 3D-gedruckten Harz ($n = 288$) sowie aus den zwei Verblendkompositen ($n = 288$) zusammen. Nach der Anwendung verschiedener Oberflächenbehandlungen (Politur, Lack, Coating) wurden die Prüfkörper für einen Zeitraum von bis zu 14 Tagen in unterschiedlichen färbenden Flüssigkeiten (Rotwein, Kurkuma, Kresse, Wasser) gelagert. Die Ermittlung der Verfärbungsrate, der Oberflächenrauigkeit sowie der freien Oberflächenenergie erfolgte zu verschiedenen Messzeitpunkten: unmittelbar nach der Oberflächenbehandlung (T_0), nach 14-tägiger Lagerung in den jeweiligen Farbmedien (T_{14}) sowie nach einer simulierten individuellen (IP) bzw. professionellen Prophylaxebehandlung (PP). Die Verfärbungsrate wurde mithilfe eines Spektrophotometers (CM-26dG, Konica Minolta) bestimmt; die Berechnung der Farbveränderungen (ΔE_{00}) zwischen den Zeitpunkten erfolgte nach der standardisierten CIEDE2000-Formel. Die freie Oberflächenenergie wurde mit dem Kontaktwinkelmesssystem Easy Drop (DSA4, Krüss) bestimmt.

Die individuelle Reinigung erfolgte mithilfe einer eigens entwickelten Zahnbürstenapparatur, bestehend aus drei in Reihe geschalteten elektrischen Zahnbürsten. Dieses System diente der Simulation einer individuellen häuslichen Mundhygiene. Bei weiterhin vorliegenden deutlichen Verfärbungen ($\Delta E_{00} > 1,8$; ISO/TR 28642) wurde zusätzlich eine professionelle Prophylaxebehandlung durchgeführt, bei der ein Prophylaxekelch in Kombination mit einer Polierpaste zum Einsatz kam.

Die erhobenen Messdaten wurden unter Anwendung nicht-parametrischer Testverfahren ausgewertet, darunter der Mann-Whitney-U-Test, der Kruskal-Wallis-Test, der Friedman-Test sowie der Wilcoxon-Test. Das Signifikanzniveau wurde auf $\alpha = 0,05$ festgelegt.

4. Ergebnisse

Nach der Politur zeigte das 3D-gedruckte Harz im Vergleich zu den beiden Verblendkompositen eine höhere Umsetzungsrate, eine rauere Oberfläche sowie einen höheren Drei-Körper-Verschleiß, jedoch geringere Martens-Parameter. Die Politur mit einem Ziegenhaarbürstchen führte bei dem 3D-gedruckten Material zu einer geringeren Biegefestigkeit im Vergleich zum Verblendkomposit VITA VM LC flow. Sowohl das Polieren mit dem Ziegenhaarbürstchen als auch die Anwendung von OPTIGLAZE color konnten die Oberflächenrauigkeit des 3D-gedruckten Harzes verringern. Die Verwendung von VITA AKZENT LC resultierte zudem in dem geringsten Drei-Körper-Verschleiß. Bei beiden Verblendkompositen erzielte die Politur mit dem Ziegenhaarbürstchen eine glattere Oberfläche, einen reduzierten Drei-Körper-Verschleiß sowie ein hohes elastisches Eindringmodul und eine hohe Biegefestigkeit. Dagegen führte der Einsatz von Silikonpolierern zumeist zu einem niedrigen elastischen Eindringmodul sowie zu einer verringerten Biegefestigkeit. Das Coating-Verfahren bewirkte ebenfalls eine Abnahme des elastischen Eindringmoduls sowie einen erhöhten Drei-Körper-Verschleiß.

Durch den Einsatz eines niedrigviskösen Lacks oder Coating-Materials wies das 3D-gedruckte Harz nach der Lagerung in den untersuchten Farbmedien (Rotwein, Kurkuma, Kresse und Wasser) geringere Verfärbungsraten auf. Die Verwendung eines Ziegenhaarbürstchens führte sowohl bei den gedruckten als auch bei den konventionell hergestellten Prüfkörpern zu einer erhöhten Farbstabilität. Unbehandelte Materialoberflächen, die als Vergleichsgruppe dienten, zeigten hingegen die höchste Disposition gegenüber den färbenden Substanzen. Die Farbmedien Rotwein und Kurkuma zeigten hohe Verfärbungspotentiale. Die Methode der Oberflächenbehandlung beeinflusste die Verfärbungsanfälligkeit insgesamt stärker als die Wahl des Materials. Die Politur der Verblendkomposite mit einem Ziegenhaarbürstchen führte zur geringsten Oberflächenrauigkeit, die unter dem klinisch relevanten Schwellenwert von $\leq 0,2 \mu\text{m}$ lag. Die individuelle Reinigung der 3D-gedruckten Prüfkörper führte zu einer Erhöhung der Oberflächenenergie, während die professionelle Prophylaxe eine Verringerung bewirkte. Hinsichtlich der Reduktion der Verfärbungsneigung erzielte die individuelle Prophylaxe bei dem Verblendkomposit GRADIA PLUS die besten Ergebnisse, während die professionelle Prophylaxe beim 3D-gedruckten Harz die höchste Reinigungswirkung zeigte.

5. Diskussion

3D-gedruckte Harze weisen derzeit im Vergleich zu konventionellen Materialien für den permanenten Zahnersatz noch geringere chemische und mechanische Eigenschaften auf. In der vorliegenden Dissertation zeigte das 3D-gedruckte Harz zwar eine hohe Umsetzungsrate (84,7–95,9 %), was mit einem geringen Restmonomergehalt und einer potenziell verbesserten Biokompatibilität assoziiert ist [11, 13, 37, 38], jedoch gleichzeitig eine erhöhte Oberflächenrauigkeit, einen stärkeren Drei-Körper-Verschleiß sowie geringere Martens-Parameter und Biegefestigkeitswerte im Vergleich zu konventionellen Verblendkompositen. Polierte Prüfkörper wiesen generell eine höhere Umsetzungsrate auf, da durch die mechanische Bearbeitung die Inhibitionsschicht entfernt werden kann. Im Gegensatz dazu erreichte das Verblendkomposit GRADIA PLUS lediglich eine Umsetzungsrate von 62,6–64,5 %, was vermutlich auf den hohen Füllstoffgehalt zurückzuführen ist [12]. Dieser wiederum trägt jedoch auch zur höheren Martens-Parameter und damit zu verbesserten mechanischen Eigenschaften bei.

Vor dem Hintergrund der noch unzureichenden Materialeigenschaften 3D-gedruckter Harze wurde in dieser Studie der Einfluss verschiedener Oberflächenbehandlungen untersucht. Bei konventionellen Verblendkompositen führte die Politur mit Ziegenhaarbürstchen zu besonders glatten Oberflächen, teils unterhalb des klinisch relevanten Schwellenwerts von 0,2 μm , wodurch bakterielle Plaqueanlagerung – und somit das Risiko für Gingivitis, Candidiasis und Parodontitis – signifikant reduziert werden kann [15]. Bei den 3D-gedruckten Harzen konnte diese Politurtechnik hingegen keine vergleichbar niedrigen Werte erzielen, was auf eine begrenzte Eignung dieser Methode für gedruckte Materialien hinweist.

Auch hinsichtlich der biaxialen Biegefestigkeit und des Drei-Körper-Verschleißes zeigten die Verblendkomposite nach Politur mit Ziegenhaarbürstchen überlegene Ergebnisse: Die Oberflächenqualität verbesserte sich durch die Reduktion von Defekten, die eine potenzielle Frakturanfälligkeit darstellen können [39], und die Abrasionsbeständigkeit war deutlich höher als bei den polierten 3D-gedruckten Harzen.

Im Vergleich verschiedener Oberflächenbehandlungen zeigten beim 3D-gedruckten Harz insbesondere Ziegenhaarbürstchen und bestimmte Lacke die besten Ergebnisse hinsichtlich der Oberflächenglätte – insbesondere im Vergleich zu unbehandelten Prüfkörpern. Dagegen führte die Politur mit Silikonpolierern oder das Coating-Verfahren zu höheren Rauigkeitswerten sowie erhöhten elastischen Eindringmodulen, was auf eine

ungünstige Veränderung der Oberflächeneigenschaften hinweist. Bezüglich des Drei-Körper-Verschleißes erwies sich die Applikation des Lacks VITA AKZENT LC als besonders effektiv: Sie resultierte in den geringsten Verschleißwerten und fungierte somit als schützende Schicht, die die Abrasion des Harzes signifikant reduzieren konnte.

Für die konventionellen Verblendkomposite galten vergleichbare Tendenzen: Die Politur mit Ziegenhaarbürstchen erzielte auch hier die besten Ergebnisse, während der Einsatz von Silikonpolierern tendenziell ungünstige Resultate lieferte.

Nach 14-tägiger Lagerung in verschiedenen Färbelösungen (Rotwein, Kurkuma, Kresse, Wasser) zeigte sich kein konsistenter Zusammenhang zwischen dem Materialtyp und dem Grad der Verfärbung. Stattdessen spielte die Art der Oberflächenbehandlung eine entscheidende Rolle.

Das 3D-gedruckte Harz zeigte ohne Oberflächenbehandlung die höchsten Verfärbungswerte, was vermutlich auf die durch den Herstellungsprozess bedingte erhöhte Oberflächenrauigkeit zurückzuführen ist. Diese Beobachtung unterstreicht die Notwendigkeit einer geeigneten Oberflächenbehandlung. Besonders wirksam erwiesen sich dabei die Anwendung von OPTIGLAZE color, das Coating-Verfahren sowie die Politur mit einem Ziegenhaarbürstchen, die zu einer deutlichen Reduktion der Verfärbung führten. Diese Wirkung kann unter anderem auf die niedrigviskösen Eigenschaften der Lacke zurückgeführt werden, die in der Lage sind, mikroskopische Porositäten auf der Materialoberfläche zu verschließen und somit die Anlagerung von Farbpigmenten zu erschweren [34].

Frühere Studien haben gezeigt, dass die Farbstabilität dentaler Restaurationsmaterialien sowohl durch die Oberflächenrauigkeit als auch durch die freie Oberflächenenergie beeinflusst wird [25-27]. Diese Zusammenhänge konnten in der vorliegenden Untersuchung insbesondere für die mit Ziegenhaarbürstchen polierten Verblendkomposite bestätigt werden. Bei diesen Materialien ging eine geringere Oberflächenrauigkeit ($\leq 0,2 \mu\text{m}$) mit einer reduzierten Verfärbungsneigung einher.

Diese Ergebnisse lassen sich damit erklären, dass Oberflächenunregelmäßigkeiten bevorzugte Anlagerungsstellen für Farbpigmente schaffen und somit die Farbaufnahme begünstigen. Im Gegensatz dazu tragen glattere Oberflächen zu einer erhöhten Farbstabilität bei [25, 27]. Für das 3D-gedruckte Harz konnte dieser Zusammenhang nur eingeschränkt nachgewiesen werden; dennoch zeigen die Resultate, dass auch hier eine

gezielte Oberflächenbearbeitung einen wesentlichen Einfluss auf die Verfärbungsresistenz nimmt.

Kurkuma verursachte aufgrund seines hohen Pigmentgehalts die stärksten Verfärbungen, gefolgt von Rotwein, dessen Säure und Polarität die Farbstoffaufnahme fördern [20-22].

GRADIA PLUS erwies sich als das am besten reinigbare Material, während das 3D-gedruckte Harz sowie VITA VM LC flow nach individueller Reinigung mit Zahnbürste und Zahnpasta deutlich stärkere Farbrückstände aufwiesen. Diese Unterschiede lassen sich vorrangig durch die höhere Oberflächenrauigkeit dieser beiden Materialien erklären, die die Anlagerung von Farbpigmenten begünstigt.

Bei stark verfärbten Prüfkörpern ($\Delta E_{00} > 1,8$) erwies sich die professionelle Reinigung mit Gummipolierern und Polierpasten als deutlich effektiver in der Entfernung von Verfärbungen – insbesondere beim 3D-gedruckten Harz, vor allem bei unbehandelten oder polierten Oberflächen. Beschichtete Prüfkörper hingegen zeigten eine geringere Reinigbarkeit.

Diese Ergebnisse sollten jedoch mit Vorsicht interpretiert werden: Beschichtete Oberflächen wiesen ursprünglich die höchsten Verfärbungswerte auf. Daraus ergibt sich ein wichtiger Grundsatz: Die Reinigbarkeit eines Materials ist nur dann relevant, wenn Verfärbungen überhaupt auftreten. Oberflächenbehandlungen sollen jedoch primär verhindern, dass es zu einer signifikanten Farbaufnahme kommt. Daher sollte die Reinigbarkeit als nachgeordneter Parameter betrachtet werden, während der Fokus auf der Prävention von Verfärbungen durch geeignete Oberflächenbehandlung liegen sollte.

Nach der individuellen Reinigung stiegen sowohl die Oberflächenrauigkeit als auch die freie Oberflächenenergie an – vermutlich durch den abrasiven Effekt der Zahnpasta. Die professionelle Reinigung – mittels Polierkelch und Polierpaste – hingegen verringerte beide Werte, da sie die Oberfläche glättete und homogener gestaltete.

6. Zusammenfassung

Ziel der vorliegenden Dissertation war es im ersten Abschnitt der Untersuchung, den Einfluss verschiedener Oberflächenbehandlungen – Lackieren, Beschichten, Polieren – auf die chemischen und mechanischen Eigenschaften eines 3D-gedruckten Harzes sowie zweier Verblendkomposite zu untersuchen. Im zweiten Teil lag der Fokus auf der Wirkung dieser Behandlungen auf die Farbstabilität, die freie Oberflächenenergie und die Oberflächenrauigkeit.

Das 3D-gedruckte Harz wies im Vergleich zu den Verblendkompositen eine höhere Umsetzungsrate auf, jedoch geringere mechanische Eigenschaften wie Martens-Parameter und Biegefestigkeit sowie eine erhöhte Oberflächenrauigkeit und stärkeren Abrieb. Die Politur mit einem Ziegenhaarbürstchen führte bei allen Materialien zu verbesserten Oberflächeneigenschaften und kann daher für alle Materialien empfohlen werden. Für das 3D-gedruckte Harz ergaben sich durch das Lackieren günstigere Werte hinsichtlich der Oberflächenrauigkeit und des Drei-Körper-Verschleißes. Die Anwendung eines Silikonpolierers sowie das Coating-Verfahren führten hingegen zu ungünstigeren Werten in den untersuchten Parametern.

Zur Reduktion von Verfärbungen erwiesen sich sowohl das Lackieren als auch das Polieren mit einem Ziegenhaarbürstchen als geeignete Maßnahmen. Die individuelle Prophylaxe hatte den größten Effekt bei GRADIA PLUS, während die professionelle Prophylaxe beim 3D-gedruckten Harz zu einer stärkeren Reduktion der Verfärbung führte. Eine Oberflächenrauigkeit von $\leq 0,2 \mu\text{m}$ wurde ausschließlich bei mit Ziegenhaarbürstchen behandelten Verblendkompositen erreicht.

7. Abstract (English)

The aim of this dissertation was, in the first part of the investigation, to examine the effects of different surface treatments – varnishing, coating, and polishing – on the chemical and mechanical properties of a 3D printed resin and two veneering composite resins. The second investigation focused on the impact of these treatments on color stability, surface free energy, and surface roughness.

Compared to the veneering composite resins, the 3D printed resin showed a higher degree of conversion but lower mechanical performance, including reduced Martens parameter and flexural strength, along with increased surface roughness and wear. Polishing with a goat hair brush improved surface properties across all materials and can be recommended for general use. For the 3D printed resin, varnishing resulted in favorable values regarding surface roughness and three-body wear. In contrast, polishing with a silicone polisher and the coating procedure led to less favorable outcomes in the evaluated parameters.

To minimize discoloration, both varnishing and goat hair brush polishing proved to be effective. Individual prophylaxis showed the greatest effect on GRADIA PLUS, while professional prophylaxis resulted in a more pronounced reduction in discoloration for the 3D printed resin. A surface roughness of $\leq 0.2 \mu\text{m}$ was only achieved in veneering composite resins polished with a goat hair brush.

RESEARCH AND EDUCATION

Impact of varnishing, coating, and polishing on the chemical and mechanical properties of a 3D printed resin and two veneering composite resins¹



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The mechanical properties of 3-dimensionally (3D) printed resins for definitive fixed dental prostheses (FDPs), including flexural strength (FS), are acceptable for single-unit FDPs.¹ However, shear bond strength and biocompatibility are lower^{2,3} compared with subtractively manufactured FDPs made from polymer-infiltrated ceramic, composite resin, or polymethyl methacrylate. For definitive FDPs, long-lasting color stability, low surface roughness, high mechanical properties, abrasion resistance,^{4,5} and a high degree of conversion (DC)^{6,7} associated with a material's biocompatibility⁸⁻¹¹ are required. The restoration's surface should be smooth and homogeneous, with the surface roughness (SR) not exceeding

ABSTRACT

Statement of problem. Three-dimensional (3D) printing enables the fast fabrication of definitive fixed dental prostheses (FDPs). However, data on the effects of surface treatments on their chemical and mechanical properties are lacking.

Purpose. The purpose of this in vitro study was to examine the influence of different surface treatments on a 3D printed resin in comparison with 2 veneering composite resins.

Material and methods. A total of 288 specimens were manufactured from a 3D printed resin (VarseoSmile Crown^{plus}) or veneering composite resins (GRADIA PLUS; VITA VM LC flow). Surfaces underwent varnishing, coating, polishing or remain untreated. Conversion rate (DC), surface roughness (SR), Martens parameter, flexural strength (FS), and 3-body wear (3BW) were determined (n=12). Statistical analysis was performed using Mann-Whitney-U, Kruskal-Wallis, and Spearman correlation tests ($\alpha=0.05$).

Results. After polishing, the 3D printed resin showed higher DC, SR, and 3BW but lower Martens parameters compared with veneering composite resins ($P<0.007$). After goat hair brushing, the 3D printed resin showed lower FS than VITA-VCR ($P=0.043$). For the 3D printed resin, goat hair brushing or GC-Varnish reduced SR, while VITA-Varnish showed the lowest 3BW ($P<0.045$). For both veneering composite resins, goat hair brushing led to low SR and 3BW and high E_{IT} and FS ($P<0.043$). Silicone polishing led to low E_{IT} of the 3D printed resin and low E_{IT} and FS of GC-VCR ($P<0.009$). Coating resulted in a lower E_{IT} than the untreated surface and higher 3BW than GC-Varnish ($P<0.030$).

Conclusions. The 3D printed resin showed higher DC, SR, 3BW and lower HM, E_{IT} , and FS values than the veneering composite resins. Polishing with a goat hair brush can be recommended for all tested materials. For the 3D printed resin, varnishing presents a promising alternative with regard to SR and 3BW. Silicone polishing and coating cannot be recommended. (J Prosthet Dent 2024;132:466.e1-e9)

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¹ Materials provided by BEGO GmbH Co KG, GC EUROPE NV, VITA Zahnfabrik H. Rauter GmbH Co KG, and Sirius Ceramics.

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Clinical Implications

The 3D printed resins for definitive restorations currently on the dental market exhibit weaker chemical and mechanical properties than traditional veneering composite resins. Therefore, surface treatments like polishing with goat hair brushing are essential to enhance their material properties.

0.2 μm to prevent plaque accumulation¹² while preventing wear of the restoration and antagonist.¹³ Previous investigations have reported that resins are prone to color instability and discoloration,^{14,15} with the discoloration rate of polymer-based materials depending on the material's composition, postprocessing, and surface treatment.¹⁶ Surface treatment by polishing, varnishing, or coating has been suggested to improve the surface quality of polymer-based restorations.^{4,12,17} Polishing is an established process, with improvements in esthetic appearance, microbial adherence to the material's surface, color stability, and mechanical properties being reported.^{12,17-25} The application of a polymerizable and low-viscosity glaze material may also enhance the surface quality of dental restorative materials and improve their properties.²⁶⁻²⁸ Previous investigations have examined the surface roughness and color stability of composite

resins after the application of varnishes, reporting a smoother and more color-stable surface than with conventional polishing.^{27,29,30} However, studies investigating varnishing or coating on 3D printed restorations are sparse.^{12,17,31,32} Therefore, the aim of the present study was to evaluate different surface treatments on the chemical and mechanical properties of a 3D printed resin and compare these results with 2 veneering composite resins, conventional materials with a history of long-term use.^{7,33} The null hypotheses were that neither the material nor the surface treatment would affect the DC, SR, Martens parameters, FS, or material loss after 3-body wear (3BW).

MATERIAL AND METHODS

Disk-shaped specimens (Ø12×1.2 ±0.2 mm) were manufactured to determine the DC, SR, Martens hardness (HM), elastic indentation modulus (E_{IT}), and FS (n=12); rectangular specimens (12×10×4 mm) were examined for 3BW (n=12) (Fig. 1). Three-dimensionally printed specimens (N=144) manufactured from photopolymerizing resin (VarseoSmile Crown^{plus}; BEGO GmbH Co KG) (Table 1) using a 3D printer (Varseo XS; BEGO GmbH Co KG) were cleaned in an ultrasonic bath (SONOREX DIGITEC DT 31H; BANDELIN electronic GmbH Co KG) with 96% ethanol (Otto Fischar GmbH Co KG) for 3 and 2 minutes and polymerized for 2×1500 flashes (Otoflash G171; NK Optik GmbH).

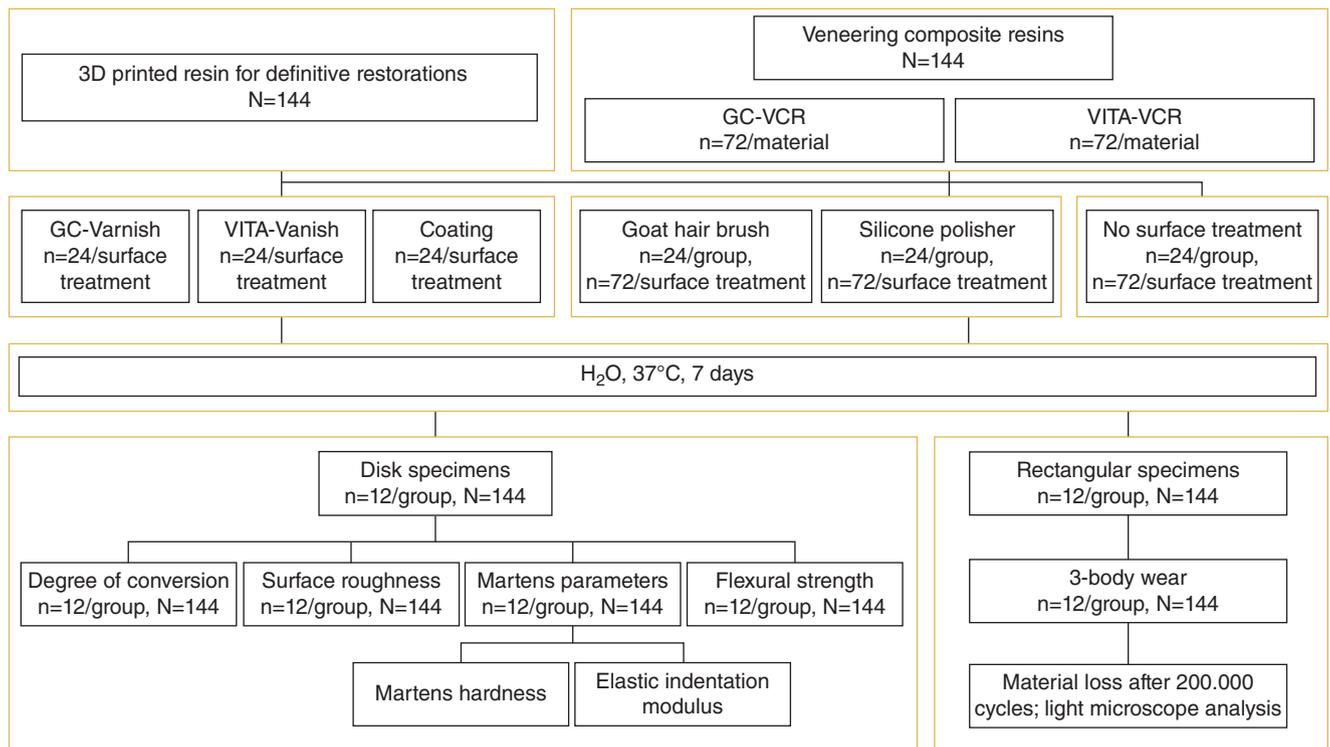


Figure 1. Study design.

Table 1. Material group, name, shade, LOT number, designation, manufacturer, and composition of materials used

Material Group	Product	Shade	LOT Number	Designation	Manufacturer	Composition (According to the Manufacturers)
3D printed resin	VarseoSmile Crown ^{plus}	A1 Dentin	600311	3D printed resin	BEGO GmbH Co KG	Esterification products of 4,4'-isopropylidendiphenol Ethoxylated and 2-methylprop-2-enoic acid Silicon oxide Methylbenzoylformate Diphenyl (2,4,6-trimethylbenzoyl) phosphine oxide Inorganic fillers (30 to 50 wt%) Barium glass (65 to 75%) Methacrylate monomer (15 to 25%) Silica (1 to 5%) Pigment (Trace) Initiator (Trace)
Veneering composite resin	GRADIA PLUS	HB-EL	211102A	GC-VCR	GC EUROPE NV	Multifunctional (meth-)acrylates (32 to 41%) Mixed oxides (silicon oxide, zirconia) (55 to 68%) Initiators and stabilizers (< 3%) Pigments (< 1%)
	VITA VM LC flow	ENAMEL	89760 98571	VITA-VCR	VITA Zahnfabrik H. Rauter GmbH Co KG	Methylmethacrylate (30%) Multifunctional acrylate (60%) Silica (10%) Photoinitiator (Trace)
Surface varnisher	OPTIGLAZE color	Clear HV	2201131	GC-Varnish	GC EUROPE NV	Methyl methacrylate and multifunctional methacrylates (30 to 40%) Urethane(meth-)acrylates (40 to 60%) Silicon oxide (8 to 11%) Ethyl-phenyl (2,4,6-trimethylbenzoyl) phosphinate (2 to 6%) Pigments < 2% other < 1%
	VITA AKZENT LC	GLAZE	89830	VITA-Varnish	VITA Zahnfabrik H. Rauter GmbH Co KG	

Veneering composite resins (GRADIA PLUS; GC EUROPE NV [GC-VCR] and VITA VM LC flow; VITA Zahnfabrik H. Rauter GmbH Co KG [VITA-VCR]) (N=144, n=72) were polymerized in 2-mm increments in silicone molds by 1 operator (M.L.) according to the manufacturer's instructions (Table 2) and ground to identical dimensions using 30- μ m-grit silicon carbide abrasive paper (Struers Waterproof Silicon Carbide Paper FEPA P#500; Struers GmbH). For experimental groups, specimens were treated using 2 different varnishes (OPTIGLAZE color; GC EUROPE NV [GC-Varnish], VITA AKZENT LC; VITA Zahnfabrik H. Rauter GmbH Co KG [VITA-Varnish]), coated with unpolymerized resin (VarseoSmile Crown^{plus}; BEGO GmbH Co KG), or polished with a goat hair brush (Rundbürste, Ziegenhaar, weiß; bredent GmbH Co KG) and polishing paste (Abraso Starglanz; bredent GmbH Co KG), or a silicone polisher (test polisher; Sirius

Ceramics). Before varnishing or coating, the specimens were airborne-particle abraded (varnished groups: 50- μ m alumina powder, 100 kPa, coated groups: 110- μ m alumina powder, 150 kPa, distance: 10 mm, angle: 45 degrees, Basic quattro; Renfert GmbH), varnished or coated, and then light polymerized. The 3D printed resin and veneering composite resins that did not undergo surface treatment acted as controls. Specimens were stored in distilled water at 37 °C for 7 days in an incubator (HeraCell 150; Kulzer GmbH).

For measuring the DC, the Raman scattering of the unpolymerized resins ($R_{\text{unpolymerized}}$) and the polymerized specimens ($R_{\text{polymerized}}$) were recorded using a Raman spectrophotometer (inVia Qontor; Renishaw GmbH). The specimens were irradiated at a wavelength of 785 nm and a spectral resolution of 1 cm^{-1} with a diode laser through a $\times 50$ microscope objective using a laser power of 100%, irradiation time of 10 seconds, and

Table 2. Technology, wavelength, polymerization time, and manufacturer of postpolymerization devices used

Name	Technology	Wavelength	Polymerization Time	Manufacturer
Otoflash G171	Flashlight, nitrogen atmosphere	Spectral range 300 to 700 nm Peaks at 480 nm and 530 nm	2x1500 flashes	NK Optik GmbH
Labolight DUO	Light-emitting diode (LED)	Spectral range 380 to 510 nm Peaks at 395 nm and 475 nm	GC-VCR: 2x3 min GC-Varnish: 90 s	GC EUROPE NV
bre.Lux PowerUnit 2	Light-emitting diode (LED)	Spectral range 370 to 500 nm	VITA-VCR: 2x360 s VITA-Varnish: 90 s	bredent GmbH Co KG

10 accumulations per run. Raman spectra were collected in the range of 1500 to 2000 cm^{-1} and analyzed by curve-fitting (WiRE 4.2 software; Renishaw GmbH). The peak heights were recorded at 1610 cm^{-1} and 1640 cm^{-1} . DC was calculated: $\text{DC} (\%) = 100 \times [1 - R_{\text{polymerized}} / R_{\text{unpolymerized}}]$, with R =band height at 1640 cm^{-1} /band height at 1610 cm^{-1} . SR was measured using a contact profilometer (MarSurf M 400; Mahr GmbH) with 3 horizontal and 3 vertical measurements with a length of 6 mm and track spacing of 0.25 mm.

The data of the arithmetic mean roughness (R_a) were recorded. To investigate HM and E_{IT} , a universal hardness testing machine (ZHU 0.2; ZwickRoell GmbH Co KG) was used.³⁴ The apex of a diamond pyramid was pressed into the specimen surface using a load of 9.8 N for 10 seconds. HM and E_{IT} were calculated on the average of 3 measurements (testX-pert V12.3 Master; ZwickRoell GmbH Co KG): $HM = \frac{F}{A_s(h)}$, with HM: Martens hardness [N/mm^2], F : test force [N], $A_s(h)$: area of the diamond indenter pyramid (26.43 for Vickers) penetrating the surface at distance h from its tip [mm^2]; $E_{IT} = (1 - \nu_s^2) \left(\frac{2\sqrt{A_p(h_c)}}{\sqrt{\pi S}} - \frac{(1 - \nu_i^2)}{E_i} \right)^{-1}$, with E_{IT} : elastic indentation modulus [kN/mm^2], $A_p(h_c)$: projected contact area under load [N/mm^2], ν_s and ν_i : Poisson ratio of specimen (0.3) and indenter, E_i : indenter's elastic modulus [N/mm^2], S : contact stiffness determined from the force removal curve. FS was determined according to DIN EN ISO 6872:2019 using the universal testing machine (Z010; ZwickRoell GmbH Co KG). Disk-shaped specimens were placed on 3 $\varnothing 3.2$ -mm steel balls forming an equilateral triangle with an edge length of 10 mm and a ball support circle of 120 degrees. With a crosshead speed of 1 mm/minute, load was applied on each specimen with a $\varnothing 1.4$ -mm centered piston until failure. The following formula was used to calculate FS: $\sigma = -0.2387 P (X - Y)/b^2$, with σ : biaxial flexural strength [MPa], P : fracture load [N], b : thickness of the specimen [mm], $X = (1 + \nu) \ln(r_2/r_3)^2 + [(1 - \nu)/2] (r_2/r_3)^2$, and $Y = (1 + \nu) [1 + \ln(r_1/r_3)^2] + (1 - \nu) (r_1/r_3)^2$, where ν : Poisson ratio, r_1 , r_2 , and r_3 : radius of the ball support, loaded area, and tested specimen [mm], b and r_3 were measured with a digital micrometer screw (Mitutoyo IP65; Mitutoyo) to a precision of 0.01 mm.

For 3BW, specimen wheels were ground using a lathe and a wear-in of 10 000 cycles (ACTA 3; SD Mechatronik GmbH). The abrasive slurry was mixed using 150 g ground millet (senegal millet; Dehner Gartencenter GmbH), 220 mL deionized water, and 0.5 g sodium azide (Merck KGaA). The specimen and antagonist stainless-steel wheel rotated in opposing directions with a 15% difference in circumferential speed, a contact pressure of 15 N, and an angular frequency of the specimen wheel of 1 Hz. The speed of the antagonistic wheel was calculated

as previously.^{35,36} The 3BW simulation was carried out for 200 000 cycles, with the abrasive medium being renewed every 50 000 cycles. A laser scanner (LAS-20; SD Mechatronik GmbH), set to a horizontal resolution of 40 μm and a vertical resolution of 0.8 μm , scanned the specimen wheels before and after simulation. Data were imported (GOM Inspect 2019; GOM GmbH) and volume loss was analyzed. The sample size of $n=12$ per group was based on similar previous studies that reported significant differences between groups for a similar or even smaller sample size.^{7,12,36,37}

All data were descriptively analyzed. Normal distribution was tested with the Kolmogorov-Smirnov test. For nonparametric analyses, Mann-Whitney U, Kruskal-Wallis, and Spearman correlation tests were performed ($\alpha=.05$). A statistical software program (IBM SPSS Statistics, v29.0; IBM Corp) was used for the analyses.

RESULTS

Nonparametric analyses were performed as all groups deviated from the normal distribution. The 3D printed resin showed a higher DC compared with GC-VCR ($P<.001$). For the 3D printed resin, silicone polishing or goat hair brushing led to higher DC than no treatment or application of VITA-Varnish ($P<.011$). Silicone polishing resulted in a higher DC than coating ($P=.001$). The application of VITA-Varnish showed the lowest DC ($P<.001$) (Fig. 2). After goat hair brushing or silicone polishing, veneering composite resins showed lower SR than the 3D printed resin ($P<.001$).

For the 3D printed resin, GC-Varnish generated lower SR than coating, no surface treatment, or polishing with a silicone polisher ($P<.045$). Goat hair brushing, VITA-Varnish, or coating resulted in lower SR compared with silicone polishing ($P<.019$). Goat hair brushing led to lower SR than no surface treatment ($P=.006$). Veneering composite resins polished with a goat hair brush led to the lowest SR, followed by silicone polishing, while no surface treatment showed the highest SR ($P<.002$) (Table 3). After goat hair brushing and no surface treatment, both veneering composite resins showed higher HM than the 3D printed resin ($P<.006$). After silicone polishing, GC-VCR showed the highest HM, followed by VITA-VCR and the 3D printed resin ($P<.007$).

For all surface treatments, the 3D printed resin showed lower E_{IT} compared with both veneering composite resins ($P<.002$). For the 3D printed resin, no treatment led to higher E_{IT} than the application of GC- and VITA-Varnish, coating, or silicone polishing ($P<.030$). The use of VITA-Varnish, coating, or polishing with a goat hair brush resulted in higher E_{IT} than silicone polishing ($P<.009$). For GC-VCR, silicone polishing resulted in the lowest E_{IT} ($P<.001$). For VITA-VCR,

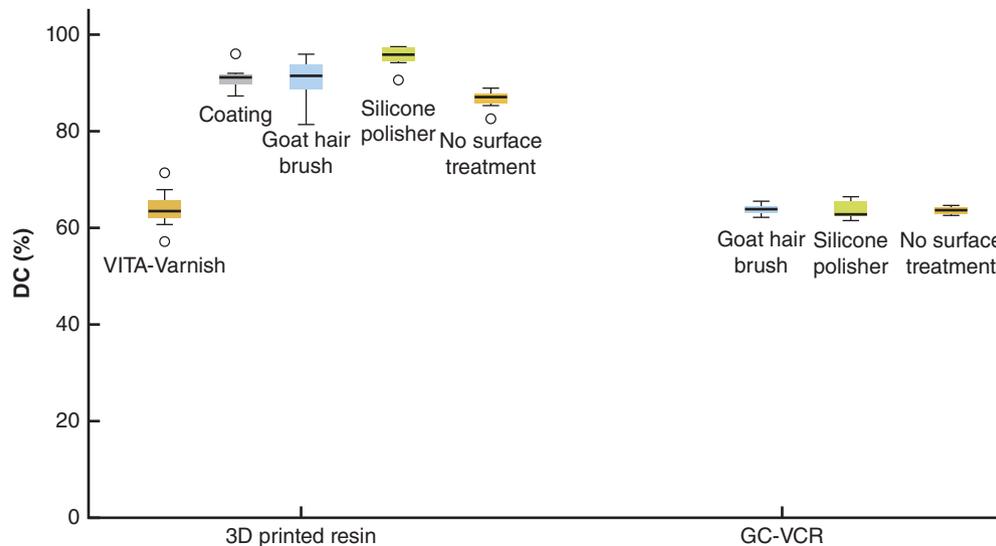


Figure 2. Degree of conversion (DC) as a percentage.

Table 3. Descriptive statistics of surface roughness R_a (μm), including median and interquartile range (IQR)

	3D Printed Resin	GC-VCR	VITA-VCR
GC-Varnish	0.303 (0.117) ^a		
VITA-Varnish	0.550 (0.185) ^{abc}		
Coating	0.558 (0.103) ^{abc}		
Goat hair brush	0.371 (0.038) ^{abB}	0.152 (0.011) ^{BA}	0.136 (0.048) ^{BA}
Silicone polisher	0.781 (0.103) ^{dB}	0.368 (0.057) ^{BA}	0.479 (0.059) ^{BA}
No surface treatment	0.615 (0.078) ^{cdA}	0.681 (0.091) ^{CA}	0.699 (0.099) ^{CA}

*Deviation from normal distribution.

Lowercase letters indicate differences between surface treatments within one material group; Uppercase letters indicate differences between materials within one surface treatment.

treatment with a goat hair brush led to the highest E_{IT} ($P<.043$) (Fig. 3). After goat hair brushing and silicone polishing, VITA-VCR showed higher FS than GC-VCR ($P<.001$). After goat hair brushing, VITA-VCR showed higher FS than the 3D printed resin ($P=.043$). For GC-VCR, silicone polishing showed the lowest FS ($P\leq.001$). For VITA-VCR, goat hair brushing led to the highest FS ($P<.001$) (Table 4).

With the spikey millet leading to inhomogeneous surfaces partly characterized by deep pits (Fig. 4), volumetric material losses are presented for 3BW (Fig. 5). After goat hair brushing or silicone polishing, both veneering composite resins showed lower 3BW than the 3D printed resin ($P<.002$). After goat hair brushing, GC-VCR led to lower 3BW than VITA-VCR ($P=.006$). For the 3D printed resin, the application of VITA-Varnish resulted in the lowest 3BW ($P<.001$), and the application of GC-Varnish led to lower 3BW than the coating technique ($P=.011$). For GC-VCR, the surface treatment with a goat hair brush showed the lowest 3BW ($P<.001$). For VITA-VCR, treatment with a silicone polisher led to lower 3BW than no surface treatment ($P=.006$). The parameters tested showed the following correlations: a positive association between HM and E_{IT} ($\rho=.841$,

$P<.001$), SR and DC ($\rho=.463$, $P=.023$), DC and 3BW ($\rho=.594$, $P<.001$) and a negative association between the Martens parameters and DC ($\rho=-.504$ /.595, $P<.001$) or 3BW ($\rho=-.275$ /.307, $P<.001$).

DISCUSSION

The aim of this investigation was to evaluate different surface treatments on the chemical and mechanical properties of a 3D printed resin and 2 veneering composite resins. While the surface treatment had no influence on HM, the hypotheses that the material or the surface treatment would not affect the DC, SR, Martens parameters, FS, or 3BW was rejected. The 3D printed resin showed a high DC (84.7 to 95.9%), indicating low residual monomer content and enhanced biocompatibility.^{8,9} These findings were consistent with those of previous investigations examining 3D printed resins for interim restorations and denture bases.^{6,10} The DC of the 3D printed resin was only compared with GC-VCR, as no peak at the aromatic C=C stretching mode could be detected for VITA-VCR. Consistent with a previous study,⁷ the DC (62.6 to 64.5%) of GC-VCR were lower than those of the 3D printed resin, attributed to the

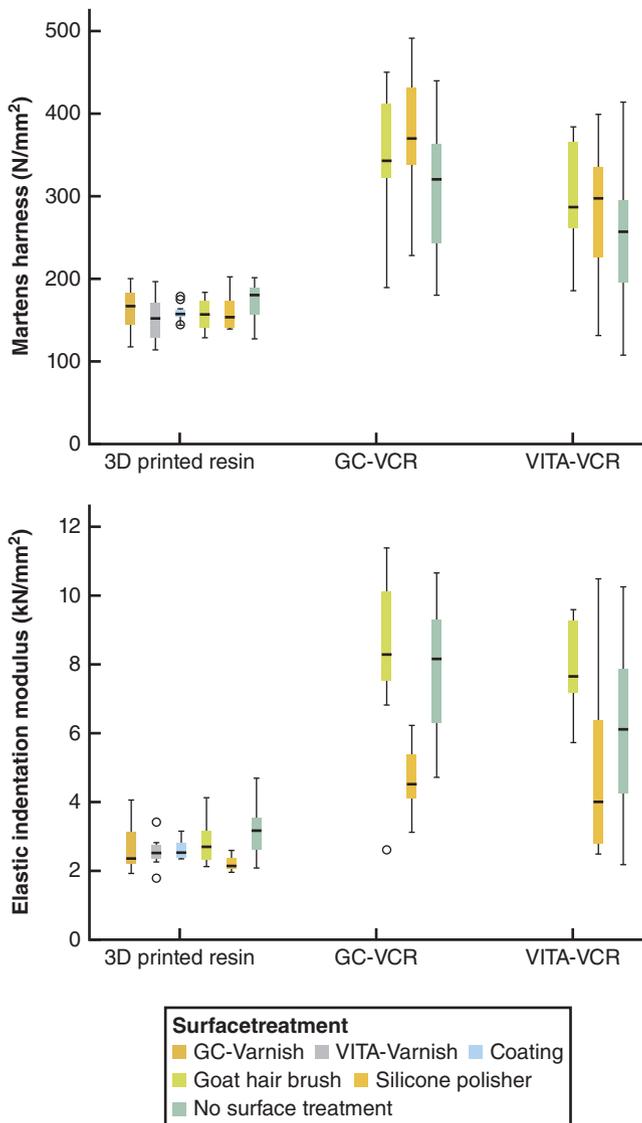


Figure 3. A, Martens hardness (N/mm^2), B, Elastic indentation modulus (kN/mm^2).

higher filler content leading to fewer monomers undergoing polymerization. While resulting in a reduced DC, the higher filler content translated into both veneering composite resins presenting higher Martens parameters and is mirrored in the negative correlation. After polishing, the 3D printed resin showed higher SR than the veneering composite resins, indicating that traditional polishing protocols are not matched to novel 3D printed resins. With goat hair brushed veneering composite resins being the only groups that showed SR below $0.2\ \mu m$, the use of the 3D printed resin for definitive restorations cannot be recommended until an improved surface treatment has been developed. After goat hair brushing, VITA-VCR showed higher FS than the 3D printed resin. Polishing did thus not only enhance the surface properties by reducing SR but

Table 4. Descriptive statistics of flexural strength FS (MPa), including median and interquartile range (IQR)

	3D Printed Resin	GC-VCR	VITA-VCR
GC-Varnish	116 (62.5) ^a		
VITA-Varnish	100 (34.6) ^a		
Coating	92.1 (45.8) ^a		
Goat hair brush	122 (47.9) ^{aA}	103 (12.0) ^{bA}	145 (15.8) ^{bB}
Silicone polisher	67.3 (79.2) ^{*aAB}	85.5 (17.2) ^{aA}	110 (28.5) ^{aB}
No surface treatment	103 (34.6) ^{aA}	103 (13.0) ^{bA}	121 (24.8) ^{aA}

*Deviation from normal distribution

Lowercase letters indicate differences between surface treatments within one material group; Uppercase letters indicate differences between materials within one surface treatment

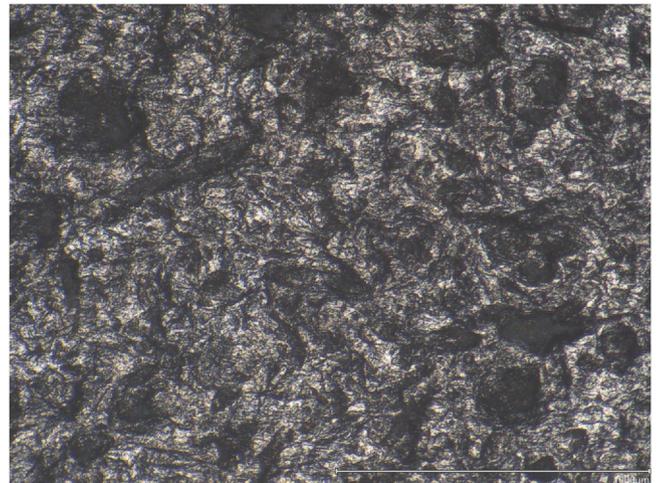


Figure 4. Inhomogeneous surface with deep pits of 3D printed resin after 200 000 cycles of 3-body wear (original magnification $\times 150$, VHX-970F; KEYENCE).

increased the mechanical properties by presumably eliminating surface faults that may act as fracture origins.²² From a clinical standpoint, for the complete and partial veneering of metal frameworks, composite resins face high masticatory forces and considerable stress and require durable flexural strength to prevent fractures.⁵ After polishing, VITA-VCR showed higher FS than GC-VCR. The lower maximum filler content in VITA-VCR may increase the flexibility and result in a higher resistance to fracture. The lower filler content in VITA-VCR could explain the lower HM after silicone polishing and the higher 3BW after goat hair brushing compared with GC-VCR. Furthermore, polished veneering composite resins presented lower 3BW than the 3D printed resin. This finding was consistent with the assumption that low surface hardness results in higher abrasion³⁵ and was confirmed by the negative correlation between Martens parameters and 3BW. Contrary to expectations, the microscopic analyses after 3BW did not show characteristic grinding marks,^{35,36} and no differences could be detected among the 3 materials for SR, FS, or 3BW if no surface treatment had been performed, underlining the importance of tailoring the surface treatment to a

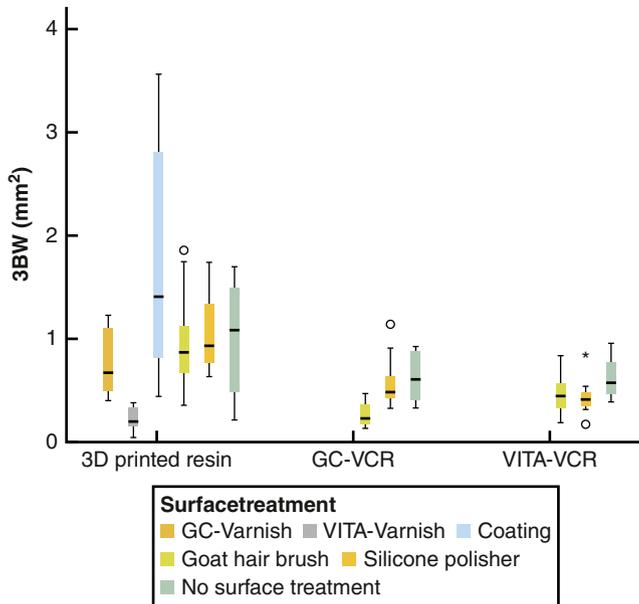


Figure 5. Three-body wear (3BW) (mm^3).

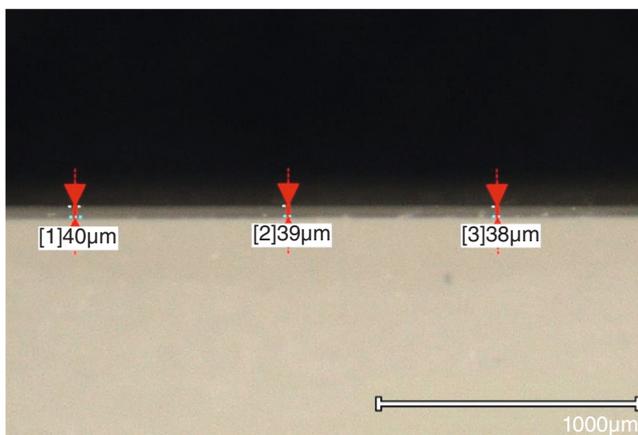


Figure 6. Layer thickness of VITA-Varnish after 200 000 cycles of three-body wear (original magnification $\times 50$) (VHX-970F; KEYENCE).

resin's mechanical properties. For the 3D printed resin, polishing resulted in a higher DC than observed after no surface treatment, which may be explained by the removal of the oxygen inhibition layer. The application of VITA-Varnish led to lower DC, which could either be related to insufficient polymerization or to the material's composition. Further research on the polymerization of varnishes is warranted, especially as the Raman spectra for GC-Varnish showed no detectable peaks.

For the 3D printed resin, surface treatment with a goat hair brush or GC-Varnish resulted in a reduced SR in comparison with no surface treatment. Different varnishes for polymer-based materials have been reported to reduce SR, in parts even under $0.2 \mu\text{m}$,^{12,28} by decreasing surface porosities through the infiltration and refilling of micropores.¹⁷ As silicone

polishing, VITA-Varnish, and coating resulted in values that were similar to without surface treatment, these protocols cannot be recommended. For the 2 veneering composite resins, both polishing protocols led to reduced SR. Goat hair brushed veneering composite resins were the only groups that presented SR below the $0.2\text{-}\mu\text{m}$ threshold, where bacterial adhesion can be prevented.¹² This favorable outcome after goat hair brushing has been reported for interim restoration materials, where a goat hair brush generated a smoother, more homogenous surface than a silicone polisher.²³ For the 3D printed resin, GC-Varnish presents a promising alternative protocol. Polishing the 3D printed resin with a silicone polisher, applying varnishes, or coating resulted in reduced E_{IT} , suggesting reduced mechanical properties. GC-VCR also showed lower E_{IT} and FS after silicone polishing. Silicone polishers can reduce a resin's mechanical properties by creating microcracks on the material surface associated with increased temperatures.^{24,25} The lower E_{IT} after coating of the 3D printed resin suggests issues with polymerization of this additional layer. Goat hair brushing yielded high E_{IT} and comparable or improved FS for both veneering composite resins, making it a recommended surface treatment. For 3BW, the application of VITA-Varnish resulted in the lowest abrasion. The layer thickness of the 2 varnishes determined in pretests ranged between 30 and $40 \mu\text{m}$. With VITA-Varnish showing a mean vertical loss of $4 \mu\text{m}$, 3BW only took place in the varnished layer, which acted as a protective coating, a finding supported by the microscopic analyses (Fig. 6). The higher abrasion for the coated specimens as well as their large scattering of 3BW results could indicate insufficient polymerization of the coating. The polymerization unit (Otoflash G171; NK Optik GmbH) used may, in contrast with polymerization during 3D printing, be unable to completely polymerize the liquid resin.¹¹ Polishing with a goat hair brush resulted in the lowest 3BW for GC-VCR, while the 2 polishing protocols showed comparable results for VITA-VCR. The smoother, more homogenous surface seems to possess a higher resistance to potential surface breakdowns²² caused by the millet. The goat hair brush can thus be recommended to decrease 3BW for both veneering composite resins.

The limitations of this investigation included the number of examined materials and surface treatments and that no a priori power analysis was performed. Post hoc power analyses compared the coated 3D printed resin with the goat hair brushed veneering composite resins. The power of a 2-sided, 2-sample *t* test exceeded 96% for a sample size of 12 specimens, with an observed effect and pooled standard deviation of 24.8% and 1.7% (DC), 0.381 and $0.046 \mu\text{m}$ (SR), 142 and 46.2N/mm^2

(HM), 5.43 and 0.907 kN/mm² (E_{IT}), 45.1 and 23.7 MPa (FS), 1.27 and 0.790 mm³ (3BW). The group selection was based on their practical significance, with polished veneering composite resins representing conventional materials with a long-term record^{7,33} and coating representing an easily implementable surface treatment for 3D printed resins requiring no further purchases and thus having the potential for widespread use. Future studies should focus on the polymerization of varnishes, as well as the esthetic properties connected to various surface treatments.

CONCLUSIONS

Based on the findings of this in vitro study, the following conclusions were drawn:

1. The 3D printed resin showed higher DC, indicating enhanced biocompatibility along with higher SR and 3BW after polishing and lower HM, E_{IT} , and FS compared with veneering composite resins. VITA-VCR showed higher FS after polishing, lower HM after silicone polishing, and higher 3BW after goat hair brushing than GC-VCR.
2. For the 3D printed resin, polishing resulted in a higher DC than observed after no surface treatment. VITA-Varnish showed a lower DC that was in the same range as observed for GC-VCR. Polishing with a goat hair brush or applying GC-Varnish resulted in a reduced SR, while VITA-Varnish showed the lowest 3BW. For the 3D printed resin, varnishing thus presents a promising alternative with regard to SR and 3BW. For both veneering composite resins, polishing with a goat hair brush led to a reduced SR, high E_{IT} and FS, and low 3BW, underscoring the significance of customized surface treatments. Polishing with a goat hair brush can be recommended for 3D printed resins and veneering composite resins. Silicone polishing led to low E_{IT} of the 3D printed resin and low E_{IT} and FS of GC-VCR. Coating resulted in a lower E_{IT} than observed for the untreated surface and higher 3BW than reported for GC-Varnish. Silicone polishing and coating can therefore not be recommended.

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RESEARCH ARTICLE OPEN ACCESS

Impact of Glazing, Coating, and Polishing on the Color Stability and Surface Properties of a 3D Printed Resin and Two Veneering Composite Resins

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ABSTRACT

Objective: To analyze the impact of various surface treatments on color stability and surface properties of a 3D printed and two veneering composite resins.

Materials and Methods: Specimens were manufactured from a 3D printed (VarseoSmile Crown^{Plus}) or two veneering composite resins (GRADIA PLUS; VITA VM LC flow) and underwent varnishing (OPTIGLAZE; VITA AKZENT LC), coating, polishing (goat hair brush; silicone polisher) or remained untreated. For 14 days, specimens were stored in red wine, curcuma, cress, or water. Individual and, for $\Delta E_{00} > 1.8$, professional prophylaxis was performed. Color (ΔE_{00}), surface free energy (SFE), and surface roughness (SR) were measured longitudinally. Mann–Whitney *U*, Kruskal–Wallis, Friedman, and Wilcoxon tests were computed ($\alpha = 0.05$).

Results: For the 3D printed resin, varnishing, coating, or goat hair brushing minimized discoloration, while untreated surfaces showed the highest discoloration. Veneering composite resins benefited from goat hair brushing. Individual and professional prophylaxis improved surface properties and partially reversed discolorations. Solely goat hair brushed veneering composite resins achieved surface roughness values $\leq 0.2 \mu\text{m}$.

Conclusions: To prevent discoloration, varnishing and goat hair brushing can be recommended for all materials. Individual prophylaxis was most effective for veneering composite resin 1, whereas professional prophylaxis significantly reduced discoloration on 3D printed resin. Only veneering composite resins treated with goat hair brushing achieved surface roughness values of $\leq 0.2 \mu\text{m}$.

Clinical Significance: As 3D printed resins tend to discolor easily, it is important to understand how different surface treatments may impact their color stability. Applying treatments such as varnishing, coating, and polishing can improve the color stability and surface properties, ensuring better esthetic results over time.

1 | Introduction

Due to recent innovations, permanent restorations can now be additively manufactured from 3D print resins and applied in a

clinical setting [1, 2]. This cost-efficient technology may represent an alternative treatment method to conventionally fabricated dental restorations, including alloy-based and ceramic restorations. Furthermore, 3D print resins can be utilized not

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only for single-tooth restorations but also as veneering materials, broadening their applications in dental prosthetics. These materials have the ability to meet the increased esthetic demands of patients who request restorations that simulate the natural tooth in color and surface texture [3]. Unfortunately, color instability and discoloration occur frequently and are reasons for replacing polymer-based restorations such as composite resins [4, 5] and 3D printed resins [6]. Previous investigations have reported a correlation between discoloration and surface roughness: high surface roughness results in an increased surface area with different predilection sites for adhesion of color pigments [7–9]. In addition to ensuring a restoration's color stability, a low surface roughness is also needed to prevent plaque accumulation. The accumulation of plaque significantly increases the patient's risk of developing gingivitis, periodontitis, or candidiasis [10, 11]. Previous studies have shown that the color stability of a restorative material depends not only on surface roughness but also on surface free energy [9, 12]. A lower contact angle results in higher surface wettability, which in turn leads to increased adhesion of color pigments and ultimately discoloration [13]. The color stability and discoloration rate of a restoration also depend on the composition of the material, the polymerization process, and the post-treatment of the restoration's surface [14]. Two significant factors causing discoloration can be identified: extrinsic factors from colorants of foods and drinks (e.g., red wine [9, 15], curcuma [16], or cress [17]) and intrinsic factors arising from chemical processes within the restorative material. These intrinsic factors can be examined by including control groups stored in distilled water and investigated over time [9, 18, 19]. Currently, the surface of 3D printed materials needs further improvement to meet the high standards of a homogenous, smooth, and color-stable surface required for long-term clinical use [20, 21]. Surface irregularities should have a smaller diameter than the average diameter of bacteria (0.2 μm), making it more difficult for them to adhere [22–24]. As of today, there are various methods to enhance the material surface after manufacturing, such as polishing, varnishing, or coating. Polymer-based restorations can be polished using diamond finishing burs, silicone polishers, goat hair brushes, impregnated disks and cups, and different polishing pastes [10, 25, 26]. Alternatively, applying low-viscosity glazing materials—a process termed varnishing—or unpolymerized 3D print resins—termed coating—to a restoration's surface can improve surface properties and result in a sealed, homogeneous, and smooth finish [26–28]. However, there is still a lack of investigations examining these effects for 3D printed materials [29, 30]. In clinical practice, discolorations of polymer-based restorations can be prevented or reversed through individual or professional prophylaxis measures. Currently, only a few investigations have explored the influence of different surface treatments on the properties of 3D printed resins after storage in various media [29, 31]. To the authors' best knowledge, the potential for cleaning and reversing discolorations has also not been sufficiently studied.

Therefore, the aim of this investigation was to examine the impact of different surface treatments—namely, varnishing, coating, polishing, or no surface treatment—on the color stability, surface free energy, and surface roughness of a 3D print and two veneering composite resins after immersion in various storage

media. Additionally, the investigation aimed to assess the potential of individual and professional prophylaxis measures to reverse any changes in these examined parameters.

The tested hypotheses were as follows:

1. Color deviation after 14 days would be the same for all materials regardless of surface treatment or storage medium; for all surface treatments regardless of material or storage medium; and for all storage media regardless of material or surface treatment.
2. Individual or professional prophylaxis could not improve color deviation, irrespective of the material, surface treatment, or storage medium.
3. Surface properties, namely surface free energy and surface roughness, would not be affected by different materials, surface treatments, storage media, or cleaning methods.

2 | Material and Methods

A total of 288 specimens were additively manufactured from photopolymerizing resin (VarseoSmile Crown^{Plus}, A1, BEGO, Bremen, Germany) using a 3D printer (Varseo XS, BEGO). The specimens were cleaned in an ultrasonic bath (SONOREX DIGITEC DT 31 H, Bandelin, Berlin, Germany) for 3 and 2 min with 96% ethanol (Otto Fischar, Saarbrücken, Germany) and then post-polymerized under a nitrogen atmosphere for 2 \times 1500 flashes (Otoflash G171, NK Optik, Baierbrunn, Germany) (Figure 1). Additionally, 288 specimens were made from two conventional veneering composite resins (GRADIA PLUS, GC EUROPE NV, Leuven, Belgium [veneering composite resin 1]; VITA VM LC flow, VITA Zahnfabrik, Bad Säckingen, Germany [veneering composite resin 2]). The veneering composite resins were placed in a silicone mold and light-cured according to the manufacturers' instructions (Table 1). Specimens were then ground to achieve dimensions of a diameter of 16 mm and a thickness of 1.2 mm using 30- μm grit silicon carbide abrasive paper (Struers Waterproof Silicon Carbide Paper FEPA P#500, Struers, Ballerup, Denmark).

After manufacturing, 3D printed specimens were subjected to various surface treatments: they were either glazed with OPTIGLAZE (GC EUROPE NV [varnish 1]) or VITA AKZENT LC (VITA Zahnfabrik [varnish 2]), coated with unpolymerized resin (VarseoSmile Crown^{Plus}, A1, BEGO), or polished using goat hair brushes (Rundbürste, Ziegenhaar, weiß, bredent, Senden, Germany) and polishing paste (Abraso Starglanz, bredent) or polished with silicone polishers (Sirius ceramics, Frankfurt, Germany). The varnishing and coating procedures were exclusively conducted on the 3D printed material, while polishing was applied to both the 3D printed resin and the veneering composite resins. Prior to varnishing or coating, the specimens underwent airborne particle abrasion (using 50/110 μm alumina powder, 100/150 kPa pressure, distance: 10 mm, angle: 45°, Basic quattro, Renfert, Hilzingen, Germany), followed by varnishing or coating and subsequent light polymerization (Table 1). Control groups consisted of specimens that did not receive any surface treatments. After post-processing, specimens were stored in distilled water at 37°C for 7 days in an incubator

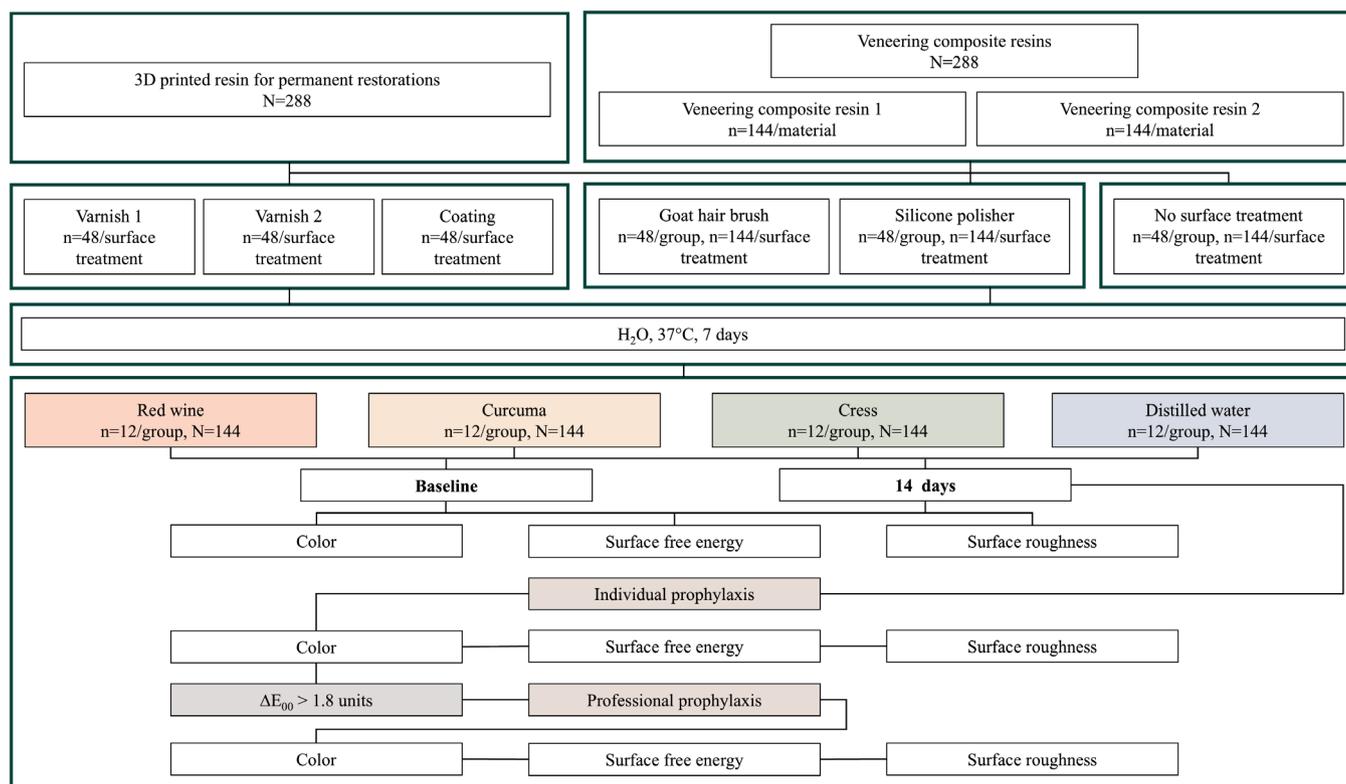


FIGURE 1 | Study design.

TABLE 1 | Name, technology, wavelength, polymerization time, and manufacturer of the polymerization devices used.

Name	Technology	Wavelength	Polymerization time	Manufacturer
Otoflash G171	Flashlight, nitrogen atmosphere	Spectral range 300–700 nm Peaks at 480 nm and 530 nm	2 × 1500 flashes	NK Optik
Labolight DUO	Light-emitting diode (LED)	Spectral range 380–510 nm Peaks at 395 nm and 475 nm	Veneering composite resin 1: 2 × 3 min Varnish 1: 90 s	GC EUROPE NV
bre.Lux PowerUnit 2	Light-emitting diode (LED)	Spectral range 370–500 nm	Veneering composite resin 2: 2 × 360 s Varnish 2: 90 s	Bredent

(HeraCell 150, Heraeus, Hanau, Germany). Measurements were then performed at baseline (T0), after 14 days (T14) of media storage at 37°C in an incubator (HeraCell 150, Heraeus), and after individual and (depending on the discoloration) professional prophylaxis.

2.1 | Media Storage

12 specimens from each group were stored in one of four different media: red wine (Cepa Lebel 2017, Lidl, Neckarsulm, Germany), curcuma (BIO KURKUMA, EWL Naturprodukte, Ransbach-Baumbach, Germany), cress (Bio Gartenkresse, EDEKA, Hamburg, Germany), or distilled water (Aqua Bidest, Kerndl, pH = 6.7, Vaterstetten, Germany). The storage

media were replaced after 7 days. For the curcuma storage medium, 40 g of curcuma powder was mixed and dissolved in 1 L of distilled water and then boiled for 10 min. The cress medium was prepared by weighing 174 g of tamped fresh cress, which was subsequently boiled with 1 L of distilled water for 10 min and then filtered through a tea strainer to remove solid particles.

2.2 | Individual Prophylaxis (IP)

After 14 days of storage in the respective media, the specimens were brushed for 4 min [9, 32] using three electronic toothbrushes (Oral-B Pulsonic Clim Clean 2000 Gray, Procter & Gamble, Schwalbach, Germany), which were connected in series in an

individually manufactured toothbrush simulator (Figure 2). To ensure that the entire surface of each specimen was adequately contacted by the bristles, the specimen holders were mounted on a movable slide rail that allowed horizontal movements at a frequency of 1 Hz. A toothpaste slurry was prepared by mixing toothpaste (Blend a Med Complete Protect Expert, Procter & Gamble) with tap water at a weight ratio of 1:2. This slurry was used during the brushing process to simulate typical oral hygiene practices.

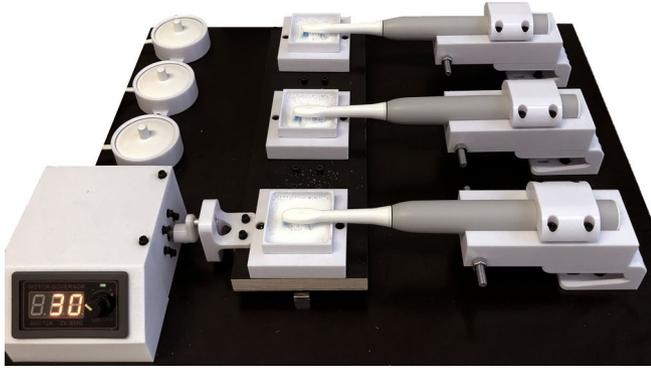


FIGURE 2 | Toothbrush simulator.

2.3 | Professional Prophylaxis (PP)

After the individual prophylaxis, measurements were conducted to assess color, surface free energy (SFE), and surface roughness (SR). If the ΔE_{00} values exceeded 1.8 (Figure 3), the specimens underwent additional cleaning for 60 s using a rubber polisher and a prophylaxis paste by hand (Cleanic, KerrHawe SA, Bioggio, Switzerland) before color, SFE, and SR measurements were repeated. In color science and dental research, the acceptability threshold (AT) is set at $\Delta E_{00} = 1.8$, according to ISO standards (ISO/TR 28642) [33], being particularly relevant in clinical practice [34–36].

2.4 | Color Measurements

After media storage, specimens were cleaned in an ultrasonic bath for 3 min in distilled water. Color measurements were conducted with a spectrophotometer CM-26dG (Konica Minolta, Tokyo, Japan). The color data software SpectraMagic NX (Konica Minolta) was used with the following parameters: standard illuminant D65, illuminating geometry d/8°, standard observer 10°, specular component included (SCI) and 100% UV. The calibration of the spectrophotometer was performed by

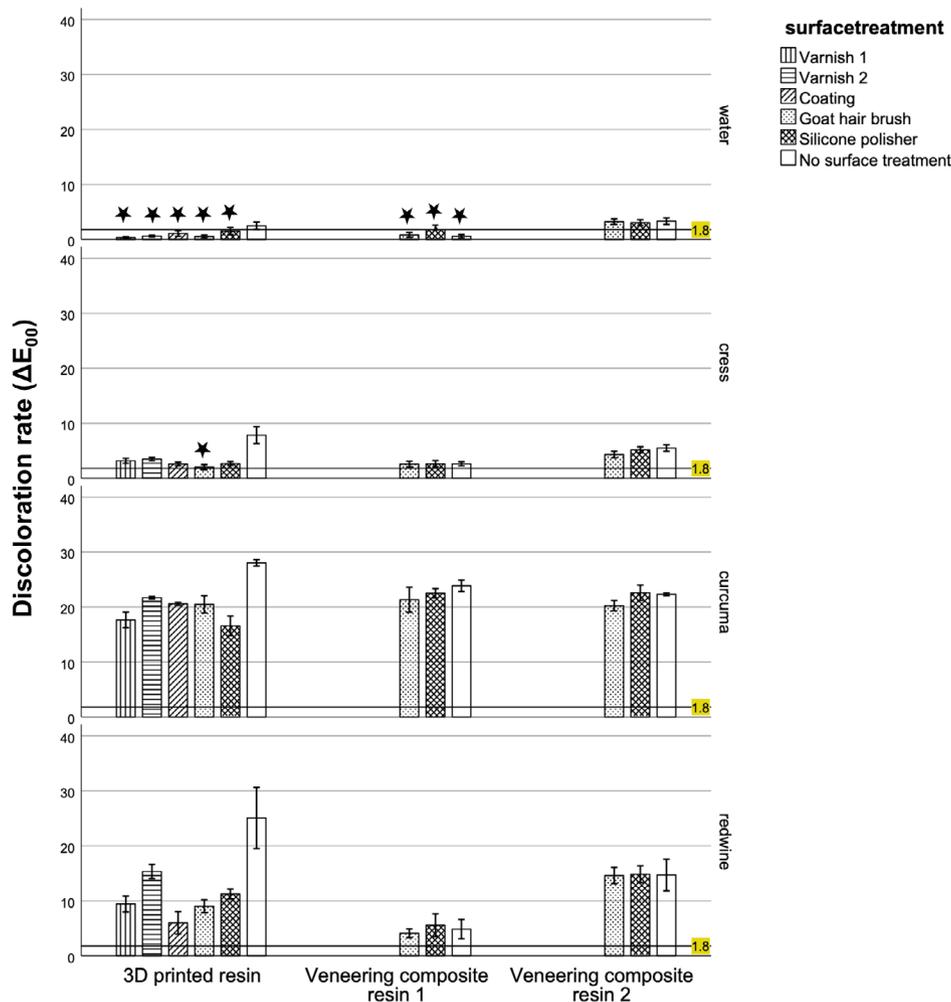


FIGURE 3 | Discoloration rates after individual prophylaxis (IP); ΔE_{00} : Baseline (T0) – IP > 1.8: Threshold for unacceptable color deviation; $\Delta E_{00} < 1.8$ is highlighted (*).

using a white tile before every measurement cycle. The average of three measurements was recorded for each specimen in front of a white and a black background. The color parameters are listed according to the CIELAB color system (Commission Internationale de l'Eclairage, CIE), whereby L^* is the lightness, a^* is the green-red, and b^* is the blue-yellow coordinate. Discolorations (ΔE_{00}) between two different time points were calculated using the standardized formula CIEDE2000 (ΔE_{00}).

$$\Delta E_{00} = \left[\left(\frac{\Delta L'}{k_L S_L} \right)^2 + \left(\frac{\Delta C'}{k_C S_C} \right)^2 + \left(\frac{\Delta H'}{k_H S_H} \right)^2 + R_T \left(\frac{\Delta C'}{k_C S_C} \right) \left(\frac{\Delta H'}{k_H S_H} \right) \right]^{1/2}$$

where $\Delta L'$, $\Delta C'$, and $\Delta H'$ represent differences in lightness, chroma, and hue; R_T reports on the interaction between chroma and hue differences in the blue region; S_L , S_C , S_H are weighting functions; K_L , K_C , K_H are parametric factors as correction terms for experimental conditions set to 1.

2.5 | Surface Free Energy

SFE analysis was performed using the drop shape analysis system Easy Drop (DSA4, Krüss, Hamburg, Germany) through the sessile drop method. Distilled water and diiodomethane (Diiodomethane, CAS No. 75-11-6, Sigma-Aldrich, Bangalore, India) were applied to each specimen at room temperature. Photographs of the specimens were taken after 5s, and the drop contour was captured using the accompanying software (Figure 4). For distilled water, the tangent-1 method was utilized, while the circular method was employed for diiodomethane. The DSA-4 software was used to determine surface tension by incorporating both disperse and polar fractions based on the measured contact angle and the properties of the liquid. Subsequently, SFE was calculated following the Owens-Wendt-Rabel-Kaelble methodology [37].

2.6 | Surface Roughness

SR was measured using a contact profilometer (MarSurf M400, Mahr, Göttingen, Germany) to determine the arithmetic mean

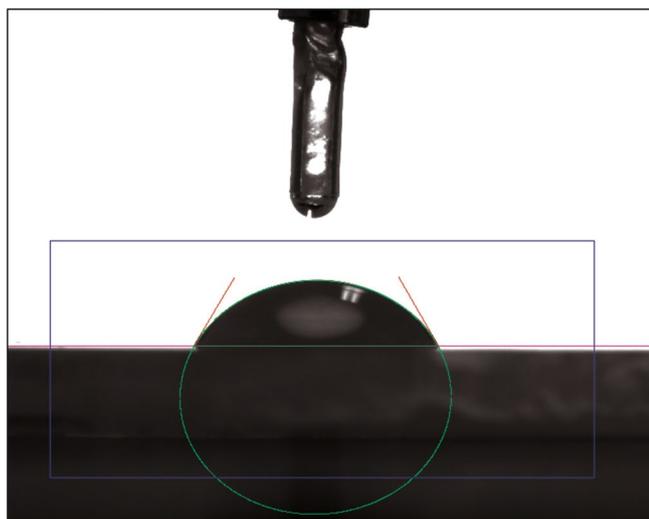


FIGURE 4 | Drop shape analysis of distilled water and diiodomethane (Easy Drop, DSA4, Krüss, Hamburg, Germany).

roughness (Ra). For each specimen, six readings were taken—three in the horizontal and three in the vertical direction. A track length of 6 mm was used for the measurements, with 0.25 mm maintained between measurement lines.

2.7 | Statistical Methods

Data underwent descriptive analysis, with normal distribution assessed via the Kolmogorov-Smirnov test. Non-parametric analyses utilized the Mann-Whitney U , Kruskal-Wallis, Friedman, and Wilcoxon tests. Significance was inferred for p values < 0.05 (IBM Statistics SPSS 29.0, IBM, Armonk, USA).

3 | Results

3.1 | Color

As 8.3% (4/48; T0-T14), 14.6% (7/48; T14-IP) and 28.2% (11/39; IP-PP) of the ΔE_{00} groups deviated from the normal distribution, non-parametric analyses were performed.

3.2 | ΔE_{00} -1: Discoloration After 14 Days Media Storage (T0-T14)

Veneering composite resin 1 showed low ΔE_{00} -1 values across various treatments and storage conditions, while veneering composite resin 2 showed high values, especially after red wine, cress, or water storage ($p < 0.001$ – 0.018). The 3D printed resin displayed variable discoloration rates, with lower ΔE_{00} -1 values after specific polishing and storage combinations, such as goat hair brushing or silicone polishing with cress or water storage ($p < 0.001$ – 0.038) (Table 2).

After red wine storage, coated 3D printed resin exhibited the least discoloration, while untreated surfaces showed the most discoloration regardless of the media ($p < 0.001$). In curcuma storage, varnish 1 minimized discoloration of the 3D printed resin ($p \leq 0.001$). The 3D printed resin showed low discoloration after cress storage with either goat hair brushing or coating ($p < 0.001$). Varnish 1 resulted in low discoloration after water storage ($p < 0.001$). For veneering composite resins 1 and 2, polishing with goat hair brushes led to low discolorations ($p < 0.001$ – 0.013).

For nearly all groups, water storage showed the lowest discoloration, followed by cress storage, and then by red wine storage, while the storage in curcuma resulted in the highest discoloration ($p < 0.001$).

3.3 | ΔE_{00} -2: Reversing Discolorations After 14 Days Media Storage by Individual Prophylaxis (T14-IP)

IP achieved high stain removal for red wine, curcuma, and cress on veneering composite resin 1, with lower discoloration removal of red wine and cress on 3D printed resin and veneering composite resin 2 ($p < 0.001$ – 0.033) (Table 3). After cress or water storage, IP maintained strong stain removal on both

TABLE 2 | Descriptive statistics (min/med/max) for ΔE_{00-1} values (T0–T14).

Material	Treatment	Red wine	Curcuma	Cress	Distilled water
3D printed resin	Varnish 1	8.59/10.5/12.3 ^{Bx}	18.9/19.8/20.1 ^{Aw}	2.92/3.66/4.03 ^{By}	0.114/0.356/0.413 ^{Az}
	Varnish 2	15.7/16.5/18.7 ^{Dx}	20.8/21.3/22.2 ^{Dw}	3.47/4.10/5.57 ^{Cy}	0.393/0.506/0.600 ^{Bz}
	Coating	4.67/6.07/9.17 ^{Ax}	20.2/20.7/21.1 ^{Cw}	2.33/2.59/3.04 ^{Ay}	0.905/1.08/2.80 ^{Dz}
	Goat hair brush	7.32/10.3/11.5 ^{Bx}	19.7/21.0/21.8 ^{bbCDw}	2.06/2.29/4.44 ^{aaAy}	0.156/0.656/1.06 ^{aCz}
	Silicone polisher	12.9/14.2/15.4 ^{bCx}	19.7/20.5/21.6 ^{abW}	3.31/3.70/4.59 ^{abY}	0.949/1.93/2.98 ^{aEz}
Veneering composite resin 1	No surface treatment	18.3/26.1/31.6 ^{efx}	26.1/26.9/27.3 ^{efx}	6.08/9.16/11.5 ^{cdY}	0.749/1.08/2.60 ^{adDz}
	Goat hair brush	5.99/7.70/8.80 ^{baX}	22.0/23.0/25.0 ^{caW}	2.68/3.38/4.40 ^{baY}	1.06/1.41/2.45 ^{baZ}
	Silicone polisher	8.15/11.9/16.3 ^{caX}	23.8/24.5/25.3 ^{cbW}	3.57/4.52/5.16 ^{bbY}	2.40/3.22/3.62 ^{bbZ}
Veneering composite resin 2	No surface treatment	7.27/9.03/13.0 ^{abX}	24.2/24.6/25.7 ^{bbW}	3.74/4.41/5.00 ^{abY}	0.413/1.33/2.09 ^{aAz}
	Goat hair brush	11.5/15.3/16.8 ^{caX}	19.0/20.2/21.5 ^{baW}	4.45/5.00/6.46 ^{caY}	2.73/3.33/4.42 ^{caZ}
	Silicone polisher	13.8/16.9/18.4 ^{cbX}	21.6/21.9/25.0 ^{bbW}	6.19/7.12/8.73 ^{cbY}	2.75/3.82/5.38 ^{caZ}
No surface treatment	11.0/16.2/23.2 ^{baBx}	21.5/22.1/22.6 ^{abW}	4.88/6.78/7.63 ^{bbY}	3.25/3.76/5.62 ^{baZ}	

Note: abc: differences between materials within one surface treatment and one medium storage; ABC: differences between surface treatments within one material group and one medium storage; zyxw: differences between media storages within one material group and one surface treatment.

TABLE 3 | Descriptive statistics (min/med/max) for ΔE_{00-2} values (T14–IP).

Material	Treatment	Red wine	Curcuma	Cress	Distilled water
3D printed resin	Varnish 1	0.842/1.07/1.52 ^{Ay}	0.991/2.28/6.82 ^{Cx}	0.789/1.01/1.21 ^{Cy}	0.077/0.107/0.262 ^{AAz}
	Varnish 2	1.12/1.56/3.10 ^{BCw}	0.322/0.712/1.76 ^{Ay}	0.749/1.10/1.89 ^{*CDx}	0.061/0.118/0.664 ^{*ABz}
	Coating	0.978/1.60/2.59 ^{Bw}	0.631/1.14/1.92 ^{Bx}	0.290/0.665/0.774 ^{Ay}	0.063/0.192/0.579 ^{BCz}
	Goat hair brush	0.899/1.71/2.16 ^{abX}	0.623/1.15/4.78 ^{aaBCx}	0.560/0.826/1.89 ^{*aBy}	0.103/0.265/0.539 ^{aCz}
	Silicone polisher	2.25/3.47/4.49 ^{bdX}	1.30/4.76/6.73 ^{bdW}	0.991/1.31/1.71 ^{aDEy}	0.187/0.468/0.993 ^{adZ}
Veneering composite resin 1	No surface treatment	1.28/2.17/4.56 ^{aCy}	4.35/5.91/8.27 ^{ceX}	1.06/1.54/2.40 ^{beZ}	0.790/1.26/2.15 ^{beZ}
	Goat hair brush	2.29/5.07/6.35 ^{baW}	2.22/3.92/4.40 ^{baX}	1.16/1.75/2.03 ^{aY}	0.326/0.760/1.39 ^{baZ}
	Silicone polisher	5.38/7.29/9.32 ^{bbW}	3.85/5.23/6.83 ^{bbX}	2.03/2.78/3.45 ^{bcY}	0.403/0.883/1.94 ^{baZ}
Veneering composite resin 2	No surface treatment	4.02/5.81/8.58 ^{caW}	3.17/4.12/5.39 ^{baX}	1.60/1.96/2.14 ^{caY}	0.477/1.15/1.59 ^{aAz}
	Goat hair brush	1.14/1.72/1.95 ^{baX}	1.00/1.78/2.39 ^{aaX}	0.807/1.28/1.68 ^{baY}	0.105/0.282/0.536 ^{aAz}
	Silicone polisher	2.30/3.46/5.57 ^{baX}	3.23/3.97/4.37 ^{*aCx}	1.71/2.17/3.08 ^{bbY}	0.419/0.843/1.40 ^{bbZ}
No surface treatment	2.26/3.15/4.85 ^{bbX}	1.84/2.91/3.93 ^{abX}	0.754/1.25/1.43 ^{aaY}	0.275/0.753/1.58 ^{abZ}	

Note: abc: differences between materials within one surface treatment and one medium storage; ABC: differences between surface treatments within one material group and one medium storage; zyxw: differences between media storages within one material group and one surface treatment.

veneering composites ($p < 0.001-0.013$), while 3D printed resin consistently showed low ΔE_{00-2} values ($p < 0.001-0.013$).

After media storage in red wine, curcuma, cress, or water, IP showed a high stain removal on the 3D-printed resin polished with a silicone polisher or left untreated ($p < 0.001-0.033$). The application of varnish 1, varnish 2, and coating led to low stain removal ($p < 0.001-0.043$). For veneering composite resin 1 and 2 polished with a silicone polisher, the IP showed a high stain removal after red wine, curcuma, or cress storage ($p < 0.001-0.028$).

For most groups, water showed the lowest removal of discoloration, followed by cress, while red wine and curcuma showed higher values ($p < 0.001-0.038$).

3.4 | ΔE_{00-3} : Reversing Persistent Discolorations After IP by Professional Prophylaxis (IP-PP)

For 39/48 groups (Figure 3), where ΔE_{00} values exceeded 1.8, indicating an unacceptable color deviation, PP was performed. For silicone polished materials, the removal of persistent red wine stains during the PP was effective on veneering composite resin 2 and the 3D printed resin ($p < 0.001-0.011$) (Table 4). For red wine, curcuma, or cress stains, the 3D printed resin and veneering composite resin 1 showed high stain removal after PP ($p < 0.001-0.073$). After water storage, the PP resulted in a high stain removal for the untreated 3D printed resin ($p < 0.001-0.007$).

After red wine storage and PP, the 3D printed resin with no surface treatment showed a high discoloration removal, followed by varnish 2 ($p < 0.001$). For curcuma stains, silicone polishing and coating resulted in a high discoloration removal, followed by no surface treatment and varnish 1 ($p < 0.001-0.003$). After PP, the stain removal of cress was promising on the untreated 3D printed resin, followed by the coated 3D printed resin ($p < 0.001-0.028$). The PP effectively removed red wine and curcuma stains on untreated or silicone-polished veneering composite resin 1, and cress stains were also best removed from untreated or goat-hair brushed surfaces ($p < 0.001-0.021$). For veneering composite resin 2, PP achieved high removal of red wine, curcuma, and cress stains, especially on untreated or silicone-polished samples ($p < 0.001-0.043$). After water storage and PP, untreated veneering composite resin 2 showed high ΔE_{00-3} values ($p = 0.002$).

For almost all tested groups, curcuma showed the highest discoloration removal during PP ($p < 0.001-0.018$).

3.5 | SFE and SR

As 6.01% (11/183) of the SFE groups and 17.5% (32/183) of the SR groups deviated from the normal distribution, nonparametric analyses were performed.

Regarding SFE, after 14 days of storage in red wine, curcuma, cress, or water, 8/48 groups showed a significant increase in SFE ($p = 0.002-0.023$), 20/48 groups exhibited a decrease

TABLE 4 | Descriptive statistics (min/med/max) for ΔE_{00-3} values (IP-PP).

Material	Treatment	Red wine	Curcuma	Cress	Distilled water
3D printed resin	Varnish 1	0.665/0.899/1.30 ^{Az}	2.56/4.33/8.05 ^{By}	0.540/0.884/1.25 ^{Az}	
	Varnish 2	3.55/4.84/8.89 ^{*Cy}	6.62/8.93/13.4 ^{Cx}	0.703/1.26/1.82 ^{Bz}	
	Coating	0.290/0.550/1.35 ^{Az}	3.16/9.14/17.1 ^{Cx}	1.04/1.83/2.94 ^{Cy}	
Veneering composite resin 1	Goat hair brush	0.421/0.941/1.82 ^{aAz}	0.737/2.08/4.46 ^{aAy}	1.11/1.92/3.47 ^{aCDz}	2.31/4.08/5.04 ^{by}
	Silicone polisher	1.32/1.93/2.60 ^{bBz}	6.24/9.82/11.9 ^{cCy}	0.997/2.51/3.66 ^{bDz}	
	No surface treatment	10.2/14.4/17.6 ^{cDx}	2.23/3.69/8.30 ^{aBy}		
	Goat hair brush	0.430/0.834/2.55 ^{*aAz}	2.06/4.33/5.74 ^{aAx}	1.14/2.07/2.42 ^{bbBy}	
Veneering composite resin 2	Silicone polisher	0.574/1.43/2.92 ^{*aBz}	5.04/6.57/7.81 ^{bBy}	0.963/1.40/3.28 ^{*aAz}	
	No surface treatment	0.899/1.30/3.38 ^{*bBz}	3.78/8.52/9.85 ^{bBx}	1.34/1.84/4.17 ^{abBy}	
	Goat hair brush	0.228/0.604/2.63 ^{aAy}	1.10/1.57/2.53 ^{*aAx}	0.623/0.999/2.85 ^{*aAy}	0.133/0.296/0.779 ^{Az}
	Silicone polisher	1.66/3.25/4.41 ^{cCx}	4.81/5.39/6.39 ^{aCw}	1.30/1.57/2.89 ^{*aBy}	0.219/0.418/1.06 ^{*aBz}
	No surface treatment	0.674/1.10/3.72 ^{*aBy}	3.30/3.67/4.57 ^{*aBw}	1.22/1.72/2.24 ^{aBx}	0.319/0.641/1.01 ^{aBz}

Note: abc: differences between materials within one surface treatment and one medium storage; ABC: differences between surface treatments within one material group and one medium storage; zyxw: differences between media storages within one material group and one surface treatment.

($p < 0.001$ – 0.031), while all other groups (20/48) showed no significant differences (Figure 5). Examining the surface treatments for 3D printed resin, 14 days of media storage led to an increase in SFE for varnish 1, varnish 2, and goat hair brushing, while silicone polishing and no surface treatment storage resulted in a decrease ($p < 0.001$ – 0.031). After IP, a total of 15/48 groups experienced a significant increase in SFE ($p < 0.001$ – 0.024), while 11/48 groups saw a decrease ($p < 0.001$ – 0.042). The IP on the surfaces of varnish 1, varnish 2, and coating resulted in an increase in SFE, whereas the IP for no surface treatment led to a decrease in SFE ($p < 0.001$ – 0.042). After PP, a total of 13/39 groups experienced an increase in SFE ($p < 0.001$ – 0.031), while 18/39 groups showed a decrease ($p < 0.001$ – 0.007). In the cases of goat hair brushing, silicone polishing, and no surface treatment, the PP resulted in a decrease in SFE ($p = 0.001$).

After 14 days of storage in various media, 6/48 groups showed a meaningful increase in SR ($p = 0.003$ – 0.042), while 12/48 groups experienced a decrease in SR ($p < 0.001$ – 0.042), and 30/48 groups showed no significant differences (Figure 6). 3D printed surfaces treated with varnish 1, goat hair brush, or no surface treatment exhibited an increase in SR after media storage ($p = 0.009$ – 0.042). In contrast, coating and silicone polishing led to a decrease in SR ($p < 0.001$ – 0.023). For veneering composite resin 1, 14 days of storage resulted in a decrease in SR for surfaces that underwent goat hair brushing, silicone polishing, and no surface treatment ($p = 0.001$ – 0.042). Following IP, 5/48 groups exhibited an increase in SR ($p = 0.001$ – 0.042), and 10/48 groups showed a decrease in SR ($p < 0.001$ – 0.032). After IP, coated 3D printed resins showed a decrease in SR ($p = 0.001$). Veneering composite resin 1 showed a decrease in SR for surfaces that were silicone polished or received no surface treatment ($p = 0.001$ – 0.017). After PP, 8/39 groups had an increase in SR ($p = 0.001$ – 0.011), whereas 13/39 groups experienced a decrease in SR ($p < 0.001$ – 0.008). Varnish 1 displayed an increase in SR, while silicone polishing and no surface treatment resulted in a decrease in SR ($p = 0.001$ – 0.008). Veneering composite resin 2 showed an increase in SR after goat hair brushing and PP, while PP led to a decrease in silicone polished or untreated surfaces ($p = 0.001$ – 0.011).

4 | Discussion

The purpose of this study was to evaluate the effects of different surface treatments (varnishing, coating, polishing, and no surface treatment) on the color stability, surface free energy, and surface roughness of a 3D printed and two veneering composite resins after immersion in various storage media. Additionally, the study aimed to determine whether individual and professional prophylaxis measures could reverse any changes observed in these parameters. As the study's findings revealed that the variables did impact color stability, surface free energy, and surface roughness in different ways depending on the conditions, the proposed hypotheses were rejected.

Although the various materials showed an impact on discoloration after 14 days of storage, the differences between the materials were not consistent. This suggests that the extent of discoloration is less influenced by the material itself and more by the type of applied surface treatment. When analyzing the

effects of surface treatment, clear trends emerged: for the 3D printed resin, varnish 1, coating, or goat hair brushing resulted in the lowest overall discoloration rates. This aligns with findings from another study where a 3D printed resin glazed with GC Optiglaze and immersed in common beverages (tea, coffee, wine) demonstrated that glazing reduces surface porosity by infiltrating the material's surface and filling micropores and defects, thereby decreasing adherence of color pigments [29]. The positive outcomes associated with polishing have also been reported in previous studies investigating color stability in both 3D print resins [30] and various composite resins [7, 12]. Polishing with aluminum-oxide or micro-diamond polishers produced the most stain-resistant surfaces. Coating emerged as a practical method since it requires no additional materials, making it preferable to options like varnishers. Another study indicated that coating is a more time- and cost-effective alternative to conventional methods such as polishing when considering surface parameters like roughness [10]. For the 3D printed resin, it was evident that not applying any surface treatment consistently led to the highest levels of discoloration, likely due to increased surface roughness resulting from the fabrication process. This highlights the importance of applying surface treatment to 3D printed surfaces. In terms of veneering composite resins, goat hair brushing resulted in the least discoloration after 14 days of storage, while silicone polishers and no treatment were less effective. The lower surface roughness achieved with goat hair brushes ($\leq 0.2 \mu\text{m}$) is likely linked to improved color stability [7, 12]. Among all tested groups, curcuma caused the most significant discoloration, followed by red wine; conversely, cress and water resulted in minimal color change. The selection of these four coloring agents was based on their established use in the literature as standard staining substances [9, 16, 17]. Foods and drinks containing pigments—such as curcuma and red wine—are known to cause pronounced staining. Curcuma's high orange pigment content leads to greater color changes compared to red wine, despite red wine's acidity and polarity enhancing surface adsorption and penetration of colorants [9, 15, 16]. Although distilled water served as a control, polymer-based materials could still experience discoloration in water due to initiator systems like camphorquinone. The results indicated that polymer-based materials such as 3D printed resins discolor at varying rates depending on the dietary factors. Therefore, it would be beneficial in future studies to include dietary information in patient history forms and consider this when selecting restorative materials.

It is recommended that patients adopt individual prophylactic measures, such as brushing their teeth with toothpaste, at least twice a day [32]. In this study, stains were most effectively removed from veneering composite resin 1, suggesting that this material is the easiest to clean using these prophylactic measures. Conversely, stains on the 3D printed resin and veneering composite resin 2 proved to be more resistant to individual cleaning efforts, likely due to the higher SR values of the 3D printed resin. The positive correlation between discoloration and surface roughness has been previously investigated and can be attributed to surface irregularities that create sites conducive to pigment adherence [7, 12]. The 3D printed resin without surface treatment or polished with a silicone polisher exhibited the highest stain removal rates during individual cleaning efforts. However, surfaces treated with glazing, such as varnishing or

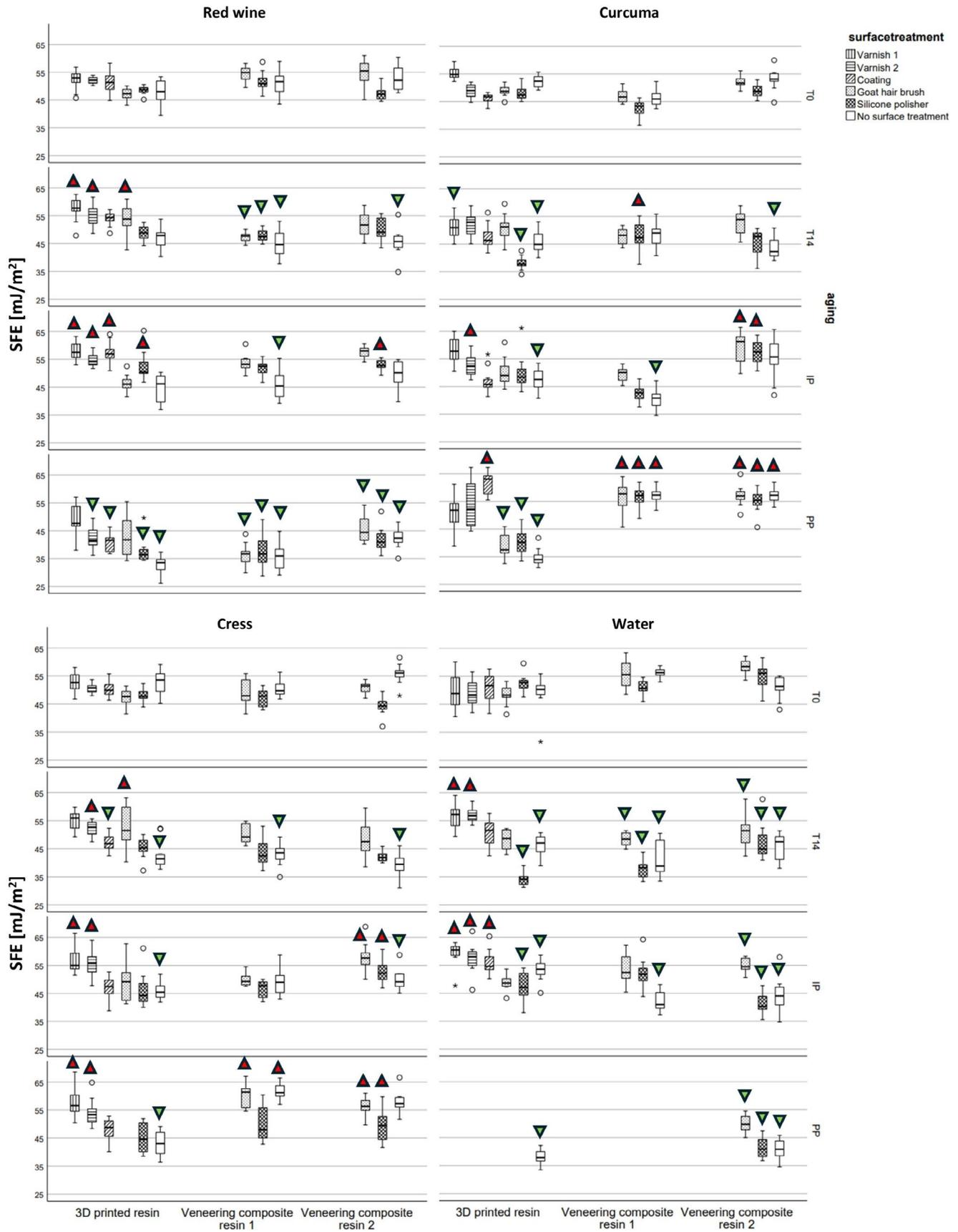


FIGURE 5 | Surface free energy (SFE) in mJ/m² at T0, T14, IP, and PP for the four different storage media for each group; ▲ SFE increases compared to T0; ▼ SFE decreases compared to T0.

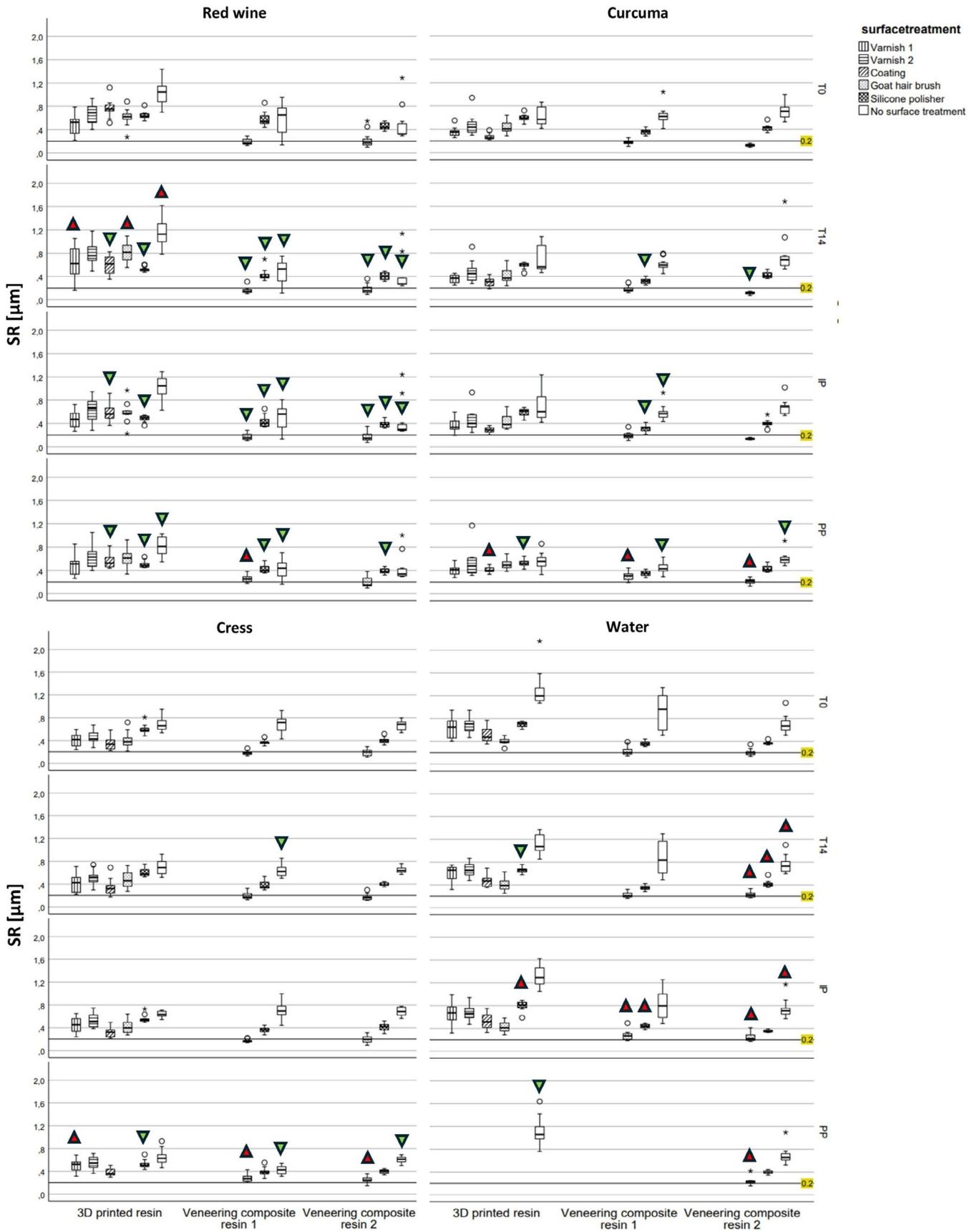


FIGURE 6 | Surface roughness (SR) in μm at T0, T14, IP, and PP for the four different storage media for each group; ▲ SFE increases compared to T0; ▼ SFE decreases compared to T0.

coating, showed the lowest stain removal effectiveness when subjected to IP. For both veneering composite resins, surfaces polished with a silicone polisher could be effectively cleaned using IP. These results should, however, be viewed with caution, as the surface-treated material should not stain significantly in the first place. The cleanability of a material should thus only constitute a parameter of secondary importance. The removal of red wine and curcuma stains during the IP was more effective than the removal of cress or water stains. For water-stored specimens, the intrinsic discoloration caused by the camphorquinone initiator system [19] could not be reversed during IP, as it cannot undo the inherent discoloration resulting from the chemical properties of the initiators within the polymers. Further studies are needed to determine why cress stains were more difficult to remove during IP than red wine or curcuma stains, even though cress groups showed overall less discoloration than red wine or curcuma.

In addition to IP measures at home, patients are advised to undergo PP measures at the dental practice at least 1 to 4 times per year [32]. PP was performed in 39 out of 48 groups (Figure 3) where ΔE_{00} values exceeded 1.8, indicating an unacceptable color deviation. In color assessment, the perceptibility threshold (PT) is set at $\Delta E_{00} = 0.8$, marking the point at which half of the observers can detect a difference, while the acceptability threshold (AT) of 1.8 is more clinically relevant, as it defines when a color difference becomes unacceptable [35, 36]. To evaluate color changes, two common formulae are used: ΔE_{ab} (CIE 76) and ΔE_{00} (CIEDE2000), as defined by ISO/TR 28642 [33]. In this study, the CIEDE2000 formula was chosen because it more accurately represents human perception, showing a stronger correlation with visual assessments (95% for ΔE_{00} vs. 75% for ΔE_{ab}) [34, 35].

The discoloration removal following PP was most effective for the 3D printed resin, followed by veneering composite resin 1. These results may be explained by the previously reported correlation between color stability, surface roughness, and surface free energy, as previous studies have shown that both surface characteristics influence the color stability of restorative materials [7, 8, 12]. In this study, the SFE and SR values for the veneering composite resins tended to increase after PP, while the values for the 3D printed resin generally decreased. Regarding the various surface treatments, untreated surfaces of the 3D printed resin were best cleaned during PP, followed by varnished (varnish 2) or coated groups. For both veneering composite resins, silicone polished or untreated surfaces were best cleaned by PP. Once again, one must consider that these surface treatments initially showed the highest discoloration rates. Through professional cleaning, stains caused by the tested beverages could, however, be partially reversed. Furthermore, studies have shown that professional cleaning can improve surface properties, such as SFE and SR, of dental materials like PEEK, PMMA, and veneering composite resins, and often help to remove discolorations [9, 32]. However, it should be noted that additional costs and time may arise for patients if they require more frequent professional cleanings that include polishing to enhance the aesthetic appearance of their prosthetic restorations.

Most of the time, the 3D printed resin showed an increase in SFE after 14 days of media storage, regardless of the surface

treatment. SFE and SR are two closely correlated surface properties. As surface roughness increases, the surface area exposed to the environment also increases, which can result in a higher SFE [11]. Similarly, smoother surfaces often exhibit lower SFE. IP also led to an increase in SFE, likely due to the roughening effect caused by toothbrushes and toothpaste, which may be attributed to the abrasive nature of the toothpaste from cleaning particles. This finding aligns with results from another study, which confirmed that cleaning methods involving toothbrushes, including sonic toothbrushes, can be recommended for cleaning polyetheretherketone (PEEK) surfaces, even though they result in increased SR values compared to manual tooth brushing [32]. In contrast, PP decreased SFE and SR, likely because the use of rubber polishers and polishing paste smooths the surface, making it more homogeneous and therefore reducing SFE, as previously reported [25]. This emphasizes the inclusion of polishing all tooth surfaces in professional prophylaxis treatments to achieve surfaces that are as clean, biofilm-free, and smooth as possible [11, 32]. Varnish 1, varnish 2, and coating resulted in higher SFE after 14 days of storage and after IP, indicating that these treatments were susceptible to surface changes during storage and the mechanical impact during IP. When comparing these results to a previous study [26], there appears to be a discrepancy in the protective effectiveness of the varnish, where varnish 2 demonstrated favorable results, with the application resulting in the lowest abrasion during three-body-wear. It was concluded that varnish 2 acted as an effective protective coating, limiting wear to just the varnished layer [26]. In this investigation, the same varnish could not offer the same level of protection when it came to maintaining SFE over time. However, surfaces treated with a goat hair brush, silicone polisher, or left untreated maintained stable SFE during storage and cleaning. Several studies have shown that polishing generally results in smoother and more homogeneous surfaces, improving the surface properties of materials [10, 12, 26]. This is also confirmed in the present study, as polishing the veneering composite resins, in some cases, even reduced roughness to below clinically relevant threshold values. This suggests that polishing methods, like those involving a goat hair brush or silicone polisher, create surfaces with fewer irregularities, which likely contributes to the stability of SFE. For the veneering composite resins, a significant increase (29/138 groups) or decrease (53/138 groups) in SFE and SR was observed, depending on the applied surface treatments. Interestingly, when no surface treatment was applied, PP reduced SR, which may explain the corresponding decrease in SFE. Further studies are necessary to understand why veneering composite resins exhibit different behaviors, particularly in relation to their surface properties and response to treatments.

It was noted that only veneering composite resins 1 and 2, when polished with a goat hair brush, consistently achieved the clinically relevant SR threshold of $\leq 0.2 \mu\text{m}$. This result was typically found across all groups, regardless of media storage, storage duration, or cleaning procedures. This finding is underlined by a previous investigation showing that goat hair brushing results in the best outcomes concerning surface roughness, Martens parameters, flexural strength, and three-body wear on veneering composite resins, indicating that this polishing system may be a better match for veneering composite resins than for 3D printed materials [26].

Previous studies have shown that the color stability of restorative materials is influenced by both surface roughness and surface free energy [7, 8, 12]. In theory, one would expect that materials with higher surface roughness and higher surface free energy would be more prone to discoloration. In this study, however, this relationship was only validated for veneering composite resins 1 and 2, which were polished with goat hair brushes. For these materials, lower surface roughness values ($\leq 0.2\mu\text{m}$) were associated with reduced discoloration rates. Further studies are needed to investigate this finding in more detail and to determine whether it applies to other materials and polishing techniques as well.

The limitations of this investigation include the number of examined materials, surface treatments, color media, and that the aging time was set to a maximum of 14 days. As a lab-based study, the conditions may not fully reflect clinical scenarios.

5 | Conclusions

Within the limitations of this current study, it was concluded that:

1. For the 3D printed resin and veneering composite resins, goat hair brushing and, in the case of 3D printed resin, varnishing effectively minimized discoloration, highlighting the clinical significance of selecting appropriate surface treatments to reduce staining, especially when exposed to agents like red wine and curcuma, which caused the most staining across all materials.
2. Stains were most effectively removed from veneering composite resin 1 with individual prophylaxis, while discoloration removal after professional prophylaxis was most effective for 3D printed resin. However, material choice, surface treatment, as well as dietary preferences, is essential, as discolorations tend to persist despite undergoing prophylaxis methods.
3. With 3D printed resin, individual prophylaxis generally increased surface free energy, while professional prophylaxis decreased it. Only goat hair brushed veneering composite resins achieved surface roughness values $\leq 0.2\mu\text{m}$, which could be relevant for long-term clinical outcomes, as smoother surfaces may reduce plaque accumulation and staining.

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Conflicts of Interest

The authors declare no conflicts of interest.

Data Availability Statement

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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**Erklärung zur Übereinstimmung der gebundenen Ausgabe der Dissertation
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Hiermit erkläre ich, dass die elektronische Version der eingereichten Dissertation mit dem Titel:

Versiegelung 3D gedruckter zahnärztlicher Restaurationen mittels Malfarbe

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