Title: The effects of pediatric dentifrices with different types of fluoride on the color change of restorative materials

Running title Staining effects of pediatric dentifrices

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Abstract

Purpose: This study aimed to evaluate the effects of dentifrices with different fluoride content on color change of restorative materials commonly used in pediatric dentistry.

Materials and methods: Three restorative materials (glass hybrid [Equia Forte (EF)], glass carbomer [GCP Glass Fill (GCP)] and compomer [Dyract XP (DXP)]) were used to prepare 120 disc shaped specimens by using a Teflon ring. Four dentifrice groups were created as Sodium Fluoride (NaF), Amine Fluoride (AmF), Stannous Fluoride (SnF₂) and no-fluoride (n = 40). Simulated tooth brushing was performed for each specimen by applying 6720 strokes for 6 months. Color changes [CIEDE2000 (ΔE_{00})] were calculated by using generalized linear model procedure and the data were subjected to two-way analysis of variance.

Results: The highest color changes for NaF and AmF dentifrice groups were observed in the GCP restorative material ($p < 0.05$). The color changes of restorative materials tested with SnF₂ dentifrice group were statistically different ($p < 0.05$) in each restorative material and ΔE_{00} values were observed as GCP > EF > DXP. SnF₂ dentifrice provided better color stability for all restorative materials when compared to NaF and AmF dentifrices; although, this was not statistically significant. GCP underwent significant discoloration values when brushed with all types of dentifrices.

Conclusion: Although the glass carbomers caused significant color change, the compomers seem to be more resistant to the color change when brushed with all types of dentifrices. The fluoride content of dentifrices is crucial for the color change of restorative materials.

Key words: pediatric dentifrices, fluorides, color change, discoloration, restorative materials

Farklı Florür İçeriğine Sahip Çocuk Diş Macunlarının Restoratif Materyallerin Renk Değişimi Üzerine Etkisi

Öz

Amaç

Bu çalışmada, farklı flor içerikli diş macunlarının pediatrik diş hekimliğinde yaygın olarak kullanılan restoratif materyallerin renk değişimi üzerindeki etkilerinin değerlendirilmesi amaçlandı.

Gereç ve yöntem

Disk şeklinde toplam 120 adet örnek hazırlamak için üç restoratif materyal (cam hibrit [Equia Forte (EF)], cam karbomer [GCP Glass Fill (GCP)] ve kompomer [Dyract XP (DXP)]) kullanıldı. Sodyum Florür (NaF), Amin Florür (AmF), Kalay Florür (SnF_2) ve florürsüz ($n = 40$) olmak üzere dört diş macunu grubu oluşturuldu. Her örnek için toplam 6720 fırça darbesi uygulanarak diş fırçalama simülasyonu gerçekleştirildi. Renk değişim değerleri CIEDE2000 (ΔE_{00}) renk sistemine göre hesaplandı ve veriler çift yönlü varyans analizine tabi tutuldu.

Bulgular: NaF ve AmF diş macunu grupları için en yüksek renk değişim değeri GCP restoratif materyalinde gözlemlendi ($p < 0.05$). SnF_2 diş macunu grubu ile test edilen restoratif materyallerin renk değişim değerleri istatistiksel olarak birbirinden farklıydı ($p < 0.05$) ve ΔE_{00} değerleri $\text{GCP} > \text{EF} > \text{DXP}$ olarak gözlemlendi. SnF_2 diş macunu, NaF ve AmF diş macunlarına kıyasla tüm restoratif materyallerde daha az renk değişimine neden oldu; ancak bu istatistiksel olarak anlamlı değildi. Tüm diş macunu gruplarında GCP en yüksek renk değişim değerleri gösterdi.

Sonuç

Tüm diş macunu gruplarında, cam karbomerler önemli renk değişimine neden olurken, kompomerlerin renk değişimine karşı daha dirençli görüldü. Diş macunlarının florid içeriği restoratif materyallerin renk değişimi için önemlidir.

Anahtar Kelimeler

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Introduction

The concept of dental aesthetics is a crucial issue for children as well as adults, since their beauty perception is affected by today's appearance-oriented culture. Maintenance of dental and oral health with better aesthetic appearance is also important for the physiological and psychological development of children (1). Various hybrid restorative materials have been developed for aesthetic and restorative purposes in pediatric dentistry including polyacid-modified composite resins (compomers), resin modified glass ionomer cements (RMGICs) and glass carbomer cement (GCP) to combine the superior properties of conventional glass ionomer cements with aesthetic advantages of composites (2). Among these restorative materials, compomers and RMGIs are widely used for restorations in pediatric dentistry due to advantageous features like fluoride release and adhesion ability (3). To improve the mechanical properties of these restorative materials, most recently glass carbomers were developed as a restorative material containing a polydialkylsiloxane component and nanofluoride hydroxyapatite particles (4).

For the success of all restorative materials, the most important criterion is the color stability after long term use. Discoloration of restorative materials can be caused by various factors as intrinsic and/or extrinsic factors (2). Intrinsic discoloration comprises staining of the restorative material itself due to the matrix type, polymer quality, amount of inorganic filler and the type of accelerator. Extrinsic discoloration arises from water absorption, water-soluble colorants adsorption, insufficient polymerization or poor oral hygiene (5). Deficient oral hygiene accelerates discoloration, since it causes accumulation of stained pellicle and colored residues. Tooth-brushing with dentifrices is widely used in home dental care to provide healthy oral hygiene. Today, there are many commercial dentifrices on the market each with a special

function. Fluorides, considered the gold standard for control and prevention of caries, have been added in dentifrices as an active ingredient in general (6).

Dentifrices have been produced with various fluoride contents such as amine fluoride (AmF), stannous fluoride (SnF₂), sodium fluoride (NaF) and sodium monofluorophosphate (SMFP) (7). Although the effects of various dentifrices with different fluoride formulations on the surface roughness of teeth and restorative materials or the effects of dentifrices on removal of tooth staining have been evaluated in several studies, the number of studies investigating the color change of restorative materials caused by the dentifrices themselves is insufficient (3, 6, 8 -13). In the present study, the following null hypotheses; 1) color change is not affected by the restorative material type, 2) there are no difference in the color stability of restorative materials after being exposed to dentifrices with different types of fluoride content were tested.

Materials and methods

Table 1 presents the characteristics of the materials evaluated in this study. In Table 2, the pediatric dentifrices with different fluoride content employed in the current study are presented.

Specimen preparation

120 specimens (8 mm in diameter × 2 mm thick) were prepared by using a Teflon ring (n = 40). The Teflon ring was covered with a strip of cellulose acetate matrix and held between two 1 mm thick glass slides to eliminate air entrapment and voids. A2 color was used in all materials to ensure standardization. The specimens were randomly divided into four dentifrices groups (n=10). G*Power software program (version 3.1.9.2; power 0.95, $\alpha = 0.05$, $\beta = 0.05$) was used to calculate the minimum sample size (10 specimens per group, n=10), based on a previous study in the literature (5).

Polymerization protocol

Polymerization of DXP specimens was carried out by using a light-emitting diode (LED) polymerization light (Elipar Free light 2, 1,200 mW/cm², 3M ESPE, Ireland) for 20 seconds to each surface, with the tip of the light on the glass slide for 40 seconds. EF restorative material was applied to each capsule with a 10-second mixer, molded with a carrier, and left at room temperature for 5 minutes to complete the hardening. According with the manufacturer's recommendation, the EF coating was applied to the surface of the specimens and cured for 20 seconds using the LED unit. GCP restorative material was applied to each capsule for 15 seconds with a mixer, molded with a carrier, and the GCP Gloss surface coating was applied following the manufacturer's guide. Curing was performed with GCP CarboLED (1,400 mW/cm² (max 60° C), GCP-Dental, Elmshorn, Germany) for 90 seconds.

Polishing and storage conditions

After polymerization, aluminum oxide discs (Sof-Lex, 3M ESPE, St. Paul, MN, USA) were used to polish each specimen sequentially with an electric hand piece, at 15,000 rpm. All specimens were numbered to identify each one and preserved in distilled water at 37° C for 24 hours.

Color change measurement and brushing cycles

The specimens were lightly rinsed and dried with tissue paper, before performing color measurement. After calibration of the clinical spectrophotometer (Vita EasyShade Advance 4.0, Ivoclar Vivadent, Liechtenstein), the color of each specimen was measured with the CIEDE2000 color system relative to D65 standard illumination against a standard white background. All measurements were repeated three times for each specimen and the mean value was calculated.

A tooth brushing simulator (Willytec, Munich, Germany) was used to mimic tooth brushing procedure. An electronic toothbrush (Oral-B Junior Kids, Procter & Gamble, USA) and soft toothbrush heads (Oral-B Sensi Ultra-thin, Procter & Gamble, USA) were used by

fixing on a holder. Each specimen was fixed on the sample holder with a standardized force of 2 N (14). Each dentifrice was diluted in distilled water in a proportion of 1:1 by weight to mimic the oral environment during tooth brushing. Considering that tooth brushing is performed twice a day, this means that each specimen will be submitted to 40 strokes in a two-minute tooth brushing, resulting total 6720 strokes (1120 strokes in a month) for 6 months. After each 1200 strokes, tooth brushes and dentifrices were renewed and this procedure was repeated for each dentifrice group (15). Specimens were removed from the sample holders, cleaned for 1 minute with an air/water spray and they were wiped with tissue paper for the final measurement. Previous studies that used water as the control group have shown that water caused no visible color change; therefore, dentifrice with no fluoride content was used instead as the control group in this study (16).

Measurement of each specimen was performed three times (L^* , c^* , h^*) with the measuring head of the spectrophotometer in accordance with the CIEDE2000 (ΔE_{00}) system. ΔE_{00} was calculated using the following formula (17) depicted in Figure 1 :

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

Figure 1. Formula used in the present study.

Evaluation of color differences was carried out ultimately via comparison with 50:50% perceptibility (PT) and 50:50% acceptability (AT) thresholds. The PT (0.81 units) and AT (1.77 units) values for CIEDE2000 (1:1:1) were obtained from a study published recently (18).

Scanning electron microscope (SEM)

After completing final measurement, randomly four specimens were selected from each group to evaluate the micro-morphology of the restorative materials by using SEM. 1,000, 2,000 and 6,000× magnifications were used to photograph the most representative areas with an accelerating voltage of 20 kV while scanning the entire surfaces.

Statistical analysis

For each variable descriptive statistics were calculated and shown as “Mean ± standard deviation (SD). Two-way ANOVA (analysis of variance) with generalized linear model procedure was used. The model included "Dentifrice", "Restorative Material" as the main effects with their two-way interaction term (Dentifrice*Restorative Material). Simple effect analysis with Bonferroni adjustment was used to break down the significant interaction effect term as post hoc analysis. A probability value of less than 0.05 was considered significant. SPSS 14.01 (SPSS Inc. Chicago, IL, USA) was used for statistical analysis.

Results

The mean color change (ΔE_{00}) and standard deviation values of all restorative materials exposed to simulated tooth brushing with four dentifrices are showed in Table 3. Statistically significant differences were indicated with superscript letters in the Table 3. The highest color change for the Brand A and B dentifrice groups was observed in GCP restorative material and this was statistically significant ($p < 0.05$). The color change for the Brand C dentifrice group was statistically different in each restorative material and ΔE_{00} values were observed as GCP>EF>DXP ($p < 0.05$). For the Brand D dentifrice group, the lowest color change was observed in the DXP material which was statistically significant ($p < 0.05$).

No significant differences were found in ΔE_{00} values of DXP restorative material brushed with different dentifrices. For EF restorative material, the highest color change was observed in the Brand D dentifrice group ($p < 0.05$). The highest color change of GCP

restorative material was observed in the Brand B dentifrice group and the lowest color change was seen in the Brand D dentifrice group ($p < 0.05$).

Evaluating the data of color change for DXP brushed with different dentifrices, it was determined that the ΔE_{00} values were lower than 1.8 (50:50% acceptability threshold value for CIEDE2000 (1:1:1) according to a recent study (18). The EF and GCP did not yield clinically acceptable ΔE_{00} values in any dentifrice group. ΔE_{00} values of three restorative materials brushed with four dentifrices are presented in Figure 2.

The SEM images ($2,000 \times$ magnifications) of the DXP, EF and GCP after simulated tooth brushing with different fluoride content dentifrices were presented in Figure 3. Similar surface features were observed with SEM analysis for the GCP and EF groups. However, the DXP group exhibited smoother surface than the other groups.

Figure 2. Color changes of the restorative materials with the dentifrices.

Figure 3. SEM images of restorative materials after simulated tooth brushing.

* a= DXP/Brand A, b=DXP/Brand B, c=DXP/Brand C, d=DXP/Brand D

e=EF/Brand A, f=EF/Brand B, g=EF/Brand C, h= EF/Brand D

i=GCP/Brand A, j=GCP/Brand B, k=GCP/Brand C, l= GCP/Brand D

Table 1. *The restorative materials used in the study and their compositions.*

Restorative material	Code	Type	Material composition	Manufacturer
Dyract XP	DXP	Poly acid-modified composite resin (Compomer)	UDMA, TEGDMA, TCB, Strontium-alumino-sodium-fluorophosphor-silicate glass SrF ₂ , SiO ₂ fillers, Filler: 73% (wt), 47% (vol), 0.8 µm	Dentsply, DeTrey, Konstanz, Germany
EQUIA Forte	EF	Glass Hybrid	Fluoro-alumino-silicate glass, Polyacrylic acid powder, Pigment, Polyacrylic acid, Distilled water, Polybasic carboxylic acid	GC, Tokyo, Japan
GCP Glass Fill	GCP	Glass carbomer	Fluoroaluminosilicate glass >90% Apatite < 6% Polyacids < 4%	GCP Dental, Ridderkerk, Netherlands

Table 2. *The dentifrices and their components.*

Dentifrices	Code	Main components	Producer
Sensodyne Pronamel Kids	Brand A	Aqua, Sorbitol, Hydrated Silica, Glycerin, PEG-6, Cocamidopropyl Betaine, Xanthan Gum, Aroma, Sodium Fluoride, Sodium Saccharin, Sucralose, Titanium Dioxide, Sodium Hydroxide, Limonene, Contains: Sodium Fluoride 0.315% w/w (1450 ppm Fluoride)	GlaxoSmithKline, Brentford, UK
Elmex Junior	Brand B	Water, sorbitol, hydrated silica, hydroxyethyl cellulose, titanium dioxides, cocamidopropyl betaines, olafluor, flavor, limonene, sodium saccharin, hydrochloride acid (1450 ppm Amine Fluoride)	GABA International AG, Therwil, Switzerland
Enamelon	Brand C	Acesulfame K, calcium/sodium maleate methyl vinyl ether copolymer, calcium sulfate, cocamidopropyl betaine, dimethicone, flavors, glycerin, lauroyl-sarcosine, monosodium phosphate, poloxamer 407, polyethylene glycol, silica, sucralose 0.45% Stannous Fluoride (1150 ppm F)	Premier Dental Products Company, PA, USA
JackNJill	Brand D	Xylitol, Purified Water, Glycerin (Coconut derived), Silica, Organic Strawberry Flavor (<i>Fragaria Chiloensis</i>), Xanthan Gum, Organic Calendula Officinalis Extract, Potassium Sorbate (Naturally derived) , Citric Acid.	JNJ Operations, Melbourne, Australia

Table 3. The mean and standard deviations of ΔE_{00} values. ^{a,b} Values in the same column with different superscripts show the statistical difference ($p < 0.05$) ^{A,B,C} Values in the same row with different superscripts show the statistical difference ($p < 0.05$).

	Restorative Material			p-Value		
	DXP	EF	GCP	Dentifrice	Material	Dentifrice*Material
	Mean $\pm$ SD	Mean $\pm$ SD	Mean $\pm$ SD			
Brand A	1.437 $\pm$ 0.56 ^{a,B}	2.302 $\pm$ 0.89 ^{b,B}	4.98 $\pm$ 1.48 ^{b,A}			
Brand B	1.758 $\pm$ 0.73 ^{a,B}	2.07 $\pm$ 1.01 ^{b,B}	6.752 $\pm$ 0.22 ^{a,A}			
				<0.001	<0.001	<0.001
Brand C	0.981 $\pm$ 0.66 ^{a,C}	2.07 $\pm$ 1.16 ^{b,B}	4.5 $\pm$ 0.35 ^{b,A}			
Brand D	1.793 $\pm$ 0.48 ^{a,B}	3.433 $\pm$ 0.77 ^{a,A}	3.055 $\pm$ 0.88 ^{c,A}			

Discussion

Main objective of this study was to evaluate the effects of dentifrices with different fluoride content on color change of restorative materials commonly used in pediatric dentistry. As the results of our study, the first null hypothesis was rejected due to the obtaining significant differences between the color changes of restorative materials after brushing with different dentifrices. There were significant differences in ΔE_{00} values of restorative materials after being exposed to dentifrices with different types of fluoride content; therefore, the second null hypothesis was rejected.

Studies generally focused on the color change caused by fluoride gels, mouthwashes and beverages (3, 19-23). While some studies have found that there was no differences of color

change between the mouthwashes and distilled water, other studies stated that the low pH of active preventive ingredients such as fluoride in the mouthwashes may affect the color stability (19, 20, 23, 24). It has been reported that fluoride varnish application negatively affects the color stability of restorative materials (25). Fatima *et al.* (26) stated that APF gel application on the GIC and RMGIC materials resulted in significant color change. While there are many studies investigating the color change caused by fluoride containing mouthwashes and local fluoride applications, the number of studies examining the color change caused by dentifrices with different fluoride formulation is very limited (1, 19, 24-27).

SnF₂ dentifrices have been implicated in surface staining because of incomplete SnF₂ stabilization ion or lack of robust cleaning ingredients (28). Liet *al.* (29) reported that SnF₂ stabilized with zinc phosphate did not induce staining and these dentifrices were very effective in stain removal of surfaces. Gerlach *et al.* (30) stated that the stabilized SnF₂/sodium hexametaphosphate dentifrices reduce the development of stain. In the present study, stabilized SnF₂ dentifrice did not cause more staining than the other dentifrices which is in agreement with the previous studies (29, 30). This may be explained with using stabilized SnF₂ dentifrice which provides some benefits without historical stannous objectionable staining.

Conforti *et al.* (27) found that a new dentifrice containing 5.0% potassium nitrate and 0.454% SnF₂ in a silica base did not cause more extrinsic dental staining than the commercially available dentifrices containing NaF. However, Artopoulou *et al.* (31) reported that the porcelain specimens that were exposed NaF showed less surface deterioration and discoloration than those were exposed SnF₂. In our study, we observed that SnF₂ dentifrice caused less color change than NaF dentifrice; however, this difference was not statistically significant.

Clinical researches have investigated the effects of dentifrices and mouth rinses containing AmF and SnF₂ on the dental plaque reduction and compared the effects of the experimental AmF/SnF₂ fluoride mouth rinses on staining (8, 32-34). West *et al.* (34) reported

that all experimental AmF/SnF₂ rinses caused more tooth staining than placebo with an overall pattern. In this study, AmF containing dentifrice caused significantly high discoloration on GCP restorative material than the other dentifrices. Since there is no study evaluating the color change caused by AmF containing dentifrice, it is not possible to make a comparison with our results.

It has been reported that tooth brushing associated with the use of dentifrices influences the optical features and surface roughness of restorative materials (15). Pires De Souza *et al.* (35) stated that since the staining susceptibility of resins is material-dependent, stains removal ability of dentifrice was not affected by the abrasives in the dentifrice. However, the results of a previous study indicated that only the dentifrices containing SnF₂ and cetylpyridinium chloride caused significant color changes for both composite materials and natural teeth (36). We found that the dentifrices used in this study caused different color changes in DXP, EF and GCP restorative materials which may be explained with the possible staining effects of the ingredients in these dentifrices.

The most common color difference system in dentistry is CIELAB, but a new color formula as CIEDE2000 (ΔE_{00}), that utilizes the concepts of chroma and hue, reinforcing the importance of the original concepts proposed by Munsell (37), has been recommended since 2001. This formula was accepted as the standard to detect color differences in 2013. Since the number of parameters used in this formula was increased, calculations became more complicated than the CIELAB formula. Color perception varies with different brightness levels according to backgrounds; this change in color perception was incorporated into the formula. Although CIELAB formula measured the distance between two points in the space basically, the addition of SL to the formula of CIE2000 had the effect of including brightness in the calculation and offers advantages by implying better clinical relevance (38). In the view of

above; ΔE_{00} was chosen to investigate the color change of dental restorative materials for this study.

Detection of color change is based on the noticeable changes in the color values of an object and the amount of color change affecting the aesthetic appearance (39). The extent of differences are defined as Perceptibility threshold (PT) and acceptability threshold (AT) as a control to evaluate the success rate of restorative materials and to interpret visual and instrumental data (18). A color change value that can be visually perceived by 50% of the observers is described as 50:50% PT and the clinically acceptable color change value for 50% of observers is described as 50:50% AT (18, 39). Therefore, color difference at or below the AT is an acceptable match in dentistry. CIEDE2000 reported 50:50% AT as 1.8 ΔE_{00} which means that $\Delta E_{00} > 1.8$ values are considered clinically unacceptable (18). Our study found that ΔE_{00} values were lower than 1.8 for only DXP among all restorative materials. This may be explained with that the more regular surface and the small particle size of DXP than the other restorative materials.

Compomer showed less color changes as compare to other groups. Greater color stability of DXP may be explained with the material's composition, as it includes hydrophilic resins, such as urethane dimethacrylate (UDMA), triethylene glycol dimethacrylate (TEGDMA) and carboxyl groups. The filler and resin particles amount affect the color resistance of restorative materials (23). Khokhar *et al.* (40) stated that urethane dimethacrylate (UDMA) content in resin matrix of materials showed lower color change than the materials with other types of dimethacrylate. The high color stability of the DXP in this study may be explained by UDMA content of the resin matrix.

In the literature, some studies have been reported that GICs are more resistant to staining because of their hydrophilic content (3, 41, 42). However, it has been stated that GICs lack color stability and stain resistance due to the degradation of metal polyacrylate salts (2, 21, 23).

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Ulusoy *et al.* (23) reported that RMGIs showed more aesthetically divergent results following the use of mouthwashes. Similarly, GCP and EF restorative materials with more glass ionomer content showed higher color change with the different pediatric dentifrices in this study. This may be explained with the large particle size of materials containing glass ionomer.

The color stability of restorative materials is also depended on its surface features. Increased surface roughness of restorative material causes water absorption through the polymer chains, influences the bonds between the matrix and filler particles resulting more staining (2, 22). In this study, significantly higher color change in GCP may be explained with its irregular surface. The irregular surface features of GCP were seen in the SEM images, as well. Besides, some cracks were observed in the SEM image of the GCP group (Figure 3). The discoloration process may be affected by these cracks.

In this study, all dentifrices were diluted in distilled water to mimic the oral environment during simulated tooth brushing. Normally, this dilution occurs in saliva which includes the enzymes, specific proteins, and ions that may decrease the effect of toothbrush abrasiveness on the specimens that may change the color stability of dental materials. Certain kind of food and beverages consumption may also cause discoloration of restorative materials in the oral environment. Limitations of this study include the possible effects of different diet and oral hygiene habits on color change of restorative materials. Considering that our results are valid for *in vitro* conditions, we believe that *in vivo* studies will reveal more comprehensive information.

Conclusion

Glass carbomers caused significant color change when brushed with all types of dentifrices. Compomers seem to be more resistant to the color change for all dentifrices used in this study. The fluoride content of dentifrices is important for the color change of restorative materials. Stannous fluoride containing dentifrice provided better color stability for all

restorative materials when compared to sodium fluoride and amine fluoride containing dentifrices.

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Author contributions: EK participated in designing the study. SY participated in generating the data for the study. EK participated in gathering the data for the study. SY participated in the analysis of the data. EK wrote the majority of the original draft of the paper. SY participated in writing the paper. SY has had access to all of the raw data of the study. EK has reviewed the pertinent raw data on which the results and conclusions of this study are based. EK, SY have approved the final version of this paper. EK, SY guarantee that all individuals who meet the Journal's authorship criteria are included as authors of this paper.

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