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Concentration and Stability of Fluoride Chemically Available in Charcoal-Containing Toothpastes

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26 **Abstract**

27 Among the wide variety of commercially oral hygiene products, activated-charcoal-based toothpastes have
28 irrupted the market, claiming multiple benefits. Although most are fluoride-free, others incorporate fluoride
29 into their formulations, yet the chemical availability and stability of fluoride in these products remain unclear.
30 Our study aimed to assess the chemical availability and stability of fluoride in commercially fluoridated
31 toothpastes formulated with activated charcoal. We purchased duplicate samples with different lot numbers
32 of different brands containing fluoride (F) and activated charcoal (n=20) in the US and Chile. Three
33 toothpastes, including sodium fluoride (NaF), sodium monofluorophosphate (MFP), and non-fluoridated
34 toothpaste, were used as controls. All toothpastes were evaluated at the time of purchase (fresh) and after
35 their expiration date (aged). We determined total-F and available total soluble-F (TSF), using a F specific
36 ion electrode. Results were expressed in mg F/kg (ppm F, w/w). The majority of evaluated toothpastes
37 contained NaF (70%) or MFP (30%) as the F salt, and all were silica-based. The TSF concentration ranged
38 from 952.6 to 1438.1 and from 925.7 to 1493.7 ppm F for fresh and aged toothpaste, respectively. TSF
39 remained close to total-F and in agreement with the F concentration reported by the manufacturer. After
40 expiring, the commercial toothpastes did not form insoluble F, except the MFP/CaCO₃- based control. In
41 conclusion, the F content in toothpastes silica-based formulated with activated charcoal is potentially
42 bioavailable and stable over time. However, concerns regarding abrasiveness, as raised by other
43 researchers, cast doubt on the suitability of these toothpastes for widespread use.

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51 Introduction

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53 Toothpastes are vehicles to deliver several compounds [1-3], including fluoride (F), which is by far the most widely
54 used and accepted active component of toothpastes to control dental caries. It is well known that F should be
55 bioavailable (chemically soluble = potentially bioavailable) in toothpastes [4], to be released into the mouth during
56 toothbrushing [5], and in concentrations of at least 1,000 ppm [6-10] to have a positive effect in the caries process [4].
57 However, due to the complexity of toothpastes formulations, the bioavailable F (also known as total soluble F [TSF]
58 or active F) might not be at the same concentration as the labeled F content [11-16]. Thus, new toothpaste
59 formulations face the challenge of maintaining F bioavailability when (new) excipients are added to the formulation
60 [1, 13, 14, 16].

61 Among new products for oral hygiene, activated charcoal-containing products have broken into the market, becoming
62 very popular in many countries across the world, with an increasingly growing market share [17, 18]. Charcoal is
63 typically a fine powder of activated charcoal [19]. Due to charcoal's capacity to abrade surfaces and adsorb
64 substances, those toothpastes are promoted to confer several positive effects such as extrinsic stain removal, tooth
65 whitening, and detoxifying properties [17]. However, those properties are debatable [17, 20, 21], and can even
66 compromise demineralization control [22] potentially contributing to tooth wear by abrasion [21, 23, 24]. Furthermore,
67 as activated charcoal-containing toothpastes are marketed as natural products, many of those are fluoride-free [25].
68 However, F salts have been added to some brands claiming an anticaries effect on the label. To the best of our
69 knowledge, there are no studies evaluating how much F is potentially bioavailable in activated-charcoal toothpastes
70 (except one brand [23]), nor their stability over time. Further, recent evidence demonstrated that total soluble fluoride
71 (TSF) determined in MFP-based toothpastes is an indicator of F chemically soluble or potentially bioavailable in
72 saliva upon toothbrushing [5]. Such information is relevant for commercial products that claim anticaries effects by
73 having F on their label. Thus, our laboratory study attempts to check the availability and stability of fluoride in those
74 new formulations.

75 Another reason to explore F concentration in those novel toothpastes, is to determine if charcoal reacts with F,
76 impacting its bioavailability. Charcoal and activated charcoal have a described high adsorption capacity due to their
77 very porous structure [18, 26, 27]. They are also able to remove fluoride from drinking water, [18, 26, 27] but in an
78 apparently low level [28]. Thus, although controversial, activated charcoal might adsorb F inside toothpastes tubes,
79 probably reducing F bioavailability. Either free fluoride ion (F⁻) from different salts such as sodium fluoride (NaF),
80 stannous fluoride (SnF₂) or amine fluoride (AmF) toothpastes formulations, or as monofluorophosphate (FPO₃²⁻), from
81 sodium monofluorophosphate (Na₂FPO₃) toothpastes, may be adsorbed by charcoal. We found only one previous

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82 study assessing F in one kind of toothpaste, that showed no adsorption of F by charcoal [23], which may not
83 represent all the other varieties of toothpaste. Furthermore, since an acidic environment also affects the adsorption
84 capacity of activated charcoal [29], we seek to determine the pH of those formulations.

85 Here we assessed the concentration of F chemically soluble found in charcoal-containing toothpastes marketed in
86 Chile and US, using a validated protocol of analysis [11] that is able to estimate fluoride bioavailability in toothpaste
87 [5, 30]. Finally, we also tested the F stability by analyzing toothpastes after exceeding its expiration period. Our
88 experimental approach measuring bioavailable F and a discussion on its use could help inform professional
89 recommendations based on evidence.

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92 **Methods**

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94 **Toothpaste Sampling**

95 Fluoridated toothpaste formulated with activated charcoal were searched in all the major drugstores and grocery
96 stores in three cities in the USA, and in Chile. The different products available on the market containing F and
97 activated charcoal were purchased in duplicate, with different lot numbers (n=2 tubes/brand). All pastes were
98 evaluated at the time of acquisition before the expiration date (fresh) and then again, after passing their expiration
99 period (aged). Thus, the F concentration of every product was analyzed fresh and aged, to evaluate F availability and
100 F stability.

101 Fresh toothpastes were stored at room temperature at 22° C until they exceeded their validity date (***natural aging***).
102 However, when those toothpastes expired, we found another four new varieties in the fresh stage. Using a validated
103 protocol [31], the four new varieties (Codes Q, R, S and T) were aged in the laboratory (to surpass their expiration
104 date described by the manufacturer) by incubation at 55°C for 16 days (equivalent to 384 hours) (***accelerated***
105 ***aging***). We also included two regular toothpastes, a sodium fluoride (NaF)/silica and a monofluorophosphate
106 (MFP)/carbonate-based abrasive toothpaste, and a negative control without fluoride. Each tube was coded for blind
107 analysis, and each tube was analyzed in duplicate.

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109 **Sample preparation for F determination**

110 Three forms of F [ionic F (IF), total soluble F (TSF) and total F (TF)] were determined according to a validated
111 protocol [11-16, 32] modified from Pearce [33]. From those analyses, the insoluble F (InsF) concentration was
112 estimated from the difference between TF and TSF concentration found in the analyses and expressed as
113 percentages calculated as: %InF=100(InsF)/(TF). MFP ion (FPO_3^{2-}) was calculated from the difference between TSF
114 and ionic fluoride [11] to confirm and estimate how much was not hydrolyzed in the brands declared as sodium
115 monofluorophosphate (Na_2FPO_3 -based).

116 In brief, 100 ± 10 mg of each toothpaste was weighed and homogenized in 10 mL of deionized water to create a
117 suspension. From that suspension, the different F fractions were obtained and measured using a selective ion-F
118 electrode. TF was obtained by mixing 0.25 mL of the suspension with 0.25 mL of 2M HCl and kept for 45 min at 45°C
119 in a water bath (Memmert GmbH+Co.KG, model WNB10, Schwabach, Germany) to induce MFP breakdown (for
120 those formulated with this salt) into ionic F and to dissolve InF. Then, the mixture was neutralized using 0.5 mL of 1M

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121 NaOH and buffered with 1 mL of TISAB II (Hanna Instruments, Woonsocket, RI, USA). To obtain TSF, the toothpaste
122 suspension was centrifuged (10 min at 3,000 g) at room temperature to remove InF bound to the abrasive. After
123 centrifugation, a 0.25 mL aliquot of the supernatant was combined with 0.25 mL of 2M HCl and maintained for 45 min
124 at 45°C to obtain TSF. Then, this solution was buffered, as described above. To obtain IF, 0.25 mL of the
125 supernatant was directly mixed with 1 mL TISAB II, 0.5 mL 1 M NaOH and 0.25 mL 2M HCl.

126 To determine F concentrations, eluents from the toothpastes were measured using a selective ion-F electrode (Orion
127 model 9609 BNWP, Orion Research, Cambridge, MA) coupled to an ion analyzer (Orion STAR A-211, Orion
128 Research, Cambridge, MA). The F electrode was first calibrated using F standards containing 0.5-32 µg F/mL. The F
129 standards were prepared with the same reagents used to analyze the samples. A linear regression equation was
130 obtained from the standards ($r^2 > 0.999$; mV vs. $\log_{10} F$). As each tube was analyzed in duplicate, 4 values were
131 obtained per each variety of toothpaste (20 different types coded from A to T; Table 1). Results were expressed as
132 ppm F (µg F/g toothpaste). Data graphics were created using GraphPad Prism (version 10.0 GraphPad Software
133 Inc.).

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135 **pH determination**

136 To determine the pH of toothpastes, two slurries from each tube were prepared. Slurries were prepared at a ratio 1:3
137 (w/v)(1 part of toothpaste [5 g] and 3 parts of de-ionized water [15 mL]). That ratio simulates the dilution that occurs
138 in the oral cavity when brushing with toothpastes [34]. Slurries were vortexed and the pH was measured using a pH
139 electrode (pH meter 9157BNMD Triode, Thermo Scientific) coupled with a potentiometer (Orion STAR A-211, Orion
140 Research). The pH electrode was initially calibrated using standard buffer solutions of pH 4, 7, and 10 (Thermo
141 Fisher Scientific™, Chelmsford, MA, USA). The electrode was recalibrated before each set of samples and
142 thoroughly washed between measurements. The mean of the pH values (n=4) was calculated for each toothpaste.
143 Data graphic was created using GraphPad Prism (version 10.0 GraphPad Software Inc.).

144 **Statistical analysis.** A descriptive analysis was carried out based on the mean and the standard deviation (SD; n=4)
145 calculated for each variety of toothpaste (two tubes analyzed in duplicate) using Excel software (Microsoft
146 Corporation). The TF concentration found was compared with the F concentration declared on the labeling by the
147 manufacturer. TSF of fresh and aged samples were also compared using a t-test using GraphPad Prism, with an α
148 error set at 5%.

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150 Results

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152 We found a total of 20 different varieties of fluoridated charcoal-containing toothpastes (Table 1) in the USA (55%;
153 n=11), and Chilean markets (45%; n=9). According to the toothpaste labeling, most toothpastes were formulated with
154 NaF (70%; n=14), while the rest were formulated with MFP (30%; n=6) (Table 1). All analyzed toothpastes declared
155 to contain silica as abrasive. Consistent with the manufacturer labeling (Table 1) TF was informed to be between
156 950–1450 ppm F (mg/F; w/w). Our analysis found that the TF ranged from 955.5 to 1471.1 and from 928.4 to 1448.5
157 ppm F for fresh and aged toothpastes, respectively (Table 2).

158 For fresh toothpastes, TSF fraction (active or bioavailable) ranged from 952.6 to 1438.1 ppm F, with similar values
159 to the TF found in all tested toothpastes. Only two toothpastes (P and S) showed TSF values that were slightly
160 below the recommended dose of 1,000 ppm F (970.5 and 952.6, respectively ppm F). Both toothpastes declared to
161 have 950 and 1000 ppm F, respectively. Overall, the pH of the fluoridated charcoal-containing toothpastes was basic
162 (Fig. 2), in a range between 6.45 to 8.24. Controls have slightly higher pH values (NaF control= 7.85; MFP control=
163 9.3) than the fluoridated charcoal-containing toothpastes.

164 For aged toothpastes, TSF fraction ranged from 925.7 to 1493.7 ppm F, with a few toothpastes with values close to
165 1000 ppm (codes I: 982.3; J:976.8; M: 991.3; N:978.9; O: 964.8; P:925.7. Comparing TSF in fresh and aged
166 toothpastes (Fig. 1), most values were not significantly reduced from fresh to aged ($p < 0.05$), except for code N and
167 the MFP control.

168 The % of InF (table 2) was almost negligible in FRESH toothpastes (3.04 ± 2.21 ; between 0-6.8%), and for AGED
169 toothpastes. The largest formation of insoluble F was for MFP/CaCO₃-base abrasive control toothpaste (29.2% of
170 Insoluble F), which was observed in both artificial and natural aging conditions.

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177 Discussion

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179 Given the increased use of fluoridated toothpastes containing activated charcoal [21], lack of information regarding its
180 availability and stability, and research indicating a lower remineralization capacity [22], we sought to experimentally
181 explore the bioavailability of F and F stability from commercial toothpastes formulations containing F and activated
182 charcoal. All evaluated toothpastes had a TSF concentration close to the F threshold for the evidence-based caries-
183 preventive effect (i.e. 1,000 ppm F) [8, 10, 14]. The F concentrations found in the tested toothpastes were consistent
184 with the US Food and Drug Administration (FDA) regulation, which establishes that toothpastes must contain at least
185 60% of its total F content as TSF [35]. We also showed that active F was maintained past its expiration time,
186 demonstrating stability overtime. Nevertheless, only the F presence in some charcoal-containing toothpaste
187 formulations is not a reason to recommend them.

188 While most commercialized toothpastes containing charcoal are fluoride-free [25], this study sought to evaluate
189 commercialized formulations containing F and charcoal. Stability was found in the concentration of F, even after
190 passing their expiration date. This is explained by its formulation, specifically the abrasive of choice, i.e. silica, which
191 is inert. We found that in all kinds of toothpaste, manufacturers declared to formulate their products either with
192 NaF/silica or MFP/silica. Since silica is an inert compound, unlike calcium-based abrasives that can react with ionic F
193 reducing the concentration of bioavailable F over time [13, 14, 16]. Silica, an inorganic filler, is unlikely to react with F
194 during storage. Thus, those formulations declared in the packages as NaF/silica or MFP/silica, are consistent with
195 our findings, where no reduction in F content was observed. As previously observed [13, 14], our MFP/CaCO₃-base
196 abrasive showed a reduction of TSF over time (around 30% is reduced after expiration).

197 Our results indicated that the incorporation of activated charcoal into fluoridated formulations does not appear to
198 affect the concentration of bioavailable F (TSF). This was not previously confirmed, because we found only one study
199 assessing F in one kind of toothpaste [23], and controversial data regarding the adsorbent properties of charcoal and
200 interaction with F [28]. Our data including twenty different varieties, analyzed in fresh and aged conditions, could
201 confirm that the amount of activated charcoal used in the tested toothpastes did not reduce the concentration of
202 active (TSF). Additionally, the pH we found on those toothpastes (from 6.45 to 8.24) is nearing neutrality, which does
203 not favor adsorption and explains the lack of interference of charcoal in the tested fluoridated charcoal-containing
204 toothpastes [18, 19, 29]. However, specific characteristics of the activated charcoal used for each formulation, and
205 even its concentration in the tube remain unknown as they are not declared by the manufacturer. We can not rule out
206 that other toothpaste formulations might have a lower pH favoring F adsorption by the charcoal, and some might vary
207 in the concentration and type of activated charcoal utilized [19], which could potentially affect F bioavailability

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208 reducing active F concentration to act in the caries process. Therefore, continuous biovigilance is always
209 recommended in novel formulations.

210 It is particularly important to consider that we only aimed at assessing the bioavailability of F in commercialized
211 toothpastes containing activated charcoal. However, a significant portion of these commercial products is F-free,
212 including brushing powders. The lack of F of such products may compromise their anticaries effect, as pointed out by
213 studies exploring other outcomes such as abrasivity [36, 37] or color change and surface properties [38]. Even having
214 F, there are other negative effects assigned to charcoal toothpastes [39]. The most reported has been the abrasivity
215 [21, 39, 40], and increased risk of tooth wear [23]. It has been mentioned that the content of charcoal had little
216 influence on the abrasive behavior observed for that toothpastes [41], but most evidence, including a systematic
217 review of *in vitro* studies, has shown that activated charcoal toothpastes are more abrasive [42]. Root surfaces may
218 be particularly susceptible to abrasiveness [43]; thus, toothbrushing with activated charcoal toothpastes should be
219 discouraged especially for older adults or adults with root surface exposure. Other negative results include an
220 increase in enamel roughness [24, 44], a reduced microhardness [44], and to generate deeper caries lesions [22].
221 Furthermore, the effectiveness of charcoal toothpastes as bleaching agents is questioned [17, 21], showing a not
222 superior effect [39, 43], or any dental bleaching effect [45]. It is important to mention that research that has found no
223 additional risk for abrasivity [36, 37] or color change and surface properties [38], does not recommend the use of
224 activated charcoal-based toothpastes use over traditional F toothpastes. Thus, the still scarce evidence on its use
225 and the potential deleterious effects associated with its use, prevent making clinical recommendations at this point.

226 Although twenty products could appear low, and a limitation of our study, we estimate that those include several
227 brands that could be purchased around the world. For example, a recent study assessing the abrasivity of charcoal
228 toothpastes tested twelve toothpastes purchased in Switzerland, and from those, only six were fluoridated [37]. From
229 those, only two brands manufactured in Spain and Switzerland were not found in the USA or Chilean markets. We
230 acquired the samples from the major retail stores in three different cities in two separate countries in America (USA,
231 in North America, and Chile, in South America). Still, it is highly possible that there are other brands and formulations
232 being sold in other countries. Thus, our results cannot be generalized to all fluoridated toothpastes containing
233 activated charcoal, and further studies testing F concentration found on those toothpastes in different regions would
234 be constantly needed. Another limitation of our study is that we were unable to find a different lot number of two
235 toothpastes (codes K and L); however, our data showed consistency between different lots. Here we did not test the
236 effect of fluoride, just if it is present (TF) and potentially active or soluble (TSF). Previous clinical evidence supports
237 that both sources of F (i.e. NaF and MFP) are effective as anticaries active [7-9]. However, we cannot assure that all
238 toothpastes having F would be equally effective. It has been shown that toothpastes with identical declared F
239 concentrations have differences in F concentrations measured intraorally, probably due to their excipients [46]. Thus,

240 it would be important to test the intraoral bioavailability of those toothpastes. Yet, to better understand the effect of
241 the different formulations, further research should test other outcomes, i.e., activity on biofilm formation with proper
242 models, onset of caries lesions, cleansing ability, and gingival irritation, among others.

243 As strengths in our study, our protocol is widely used and has been validated to study bioavailable F in toothpastes
244 [11-16]. All samples were analyzed blindly and in duplicate. In addition, we tested with a pilot study that the
245 centrifugation process is effective in removing the insoluble F adhered to the abrasive or activated charcoal (data not
246 shown). The analysis carried out for the fresh condition was done at the purchase moment (before the expiration date
247 informed by the manufacturer). The aged condition was measured after the expiration date informed by the
248 manufacturer. It's important to remark that we profusely searched the market in two countries, one in North America
249 (U.S.A.) and the other in South America (Chile). When the toothpastes aged naturally we found 4 new toothpastes,
250 and they were included. To analyze them in an aged state, we used a validated protocol (accelerated aging) [31], so
251 the total number of samples increased by 25% (from 16 to 20).

252 We cannot avoid remarking that our data has a direct clinical implication. Daily habits such as hygiene routine
253 strongly impact oral and dental health, and deciding on which product to use for brushing has paramount relevance.
254 However, it has been reported that scarce information about charcoal-based products explains the low awareness
255 and knowledge about their use and potentially adverse effects of these products among dental professionals [47].
256 Therefore, dentists and patients need to be informed about commercial products, especially new formulations such
257 as those evaluated in this study. Based on the still scarce evidence on this topic, the uncertain effectiveness against
258 dental caries, and some additional potential risks (i.e., tooth wear), these products should not be recommended to
259 replace the already tested F formulations that are being used by the patients. When asked, dentists should inform
260 patients that activated charcoal toothpastes lack scientific evidence and should not replace conventional F-containing
261 toothpastes with at least 1,000 ppm F until more research is available. In other words, these toothpastes should not
262 be recommended by dental professionals.

263 In conclusion, our findings indicated that the incorporation of activated charcoal into fluoridated formulations does not
264 decrease the concentration of soluble F available and apparently does not decrease its stability over time. However,
265 despite having bioavailable F in the threshold to act against dental caries, there are other properties that were not
266 evaluated in this study, and that could be detrimental to oral health care, such abrasiveness and disadvantages
267 related to tooth wear in charcoal toothpastes [17, 21, 23, 43, 48].

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276

277 **Conflict of Interest Statement**

278 The authors declare to have any commercial or financial relationships construed as a potential conflict of interest.

279

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283

284 **Author Contributions**

285 CEF contributed to the conception, design, data acquisition, analysis, and interpretation, drafted and critically revised
286 the manuscript; NGM contributed to data acquisition, analysis, and interpretation, drafted and critically revised the
287 manuscript; JAC and DZ contributed to data analysis and interpretation, and critically revised the manuscript. All
288 authors revised and approved the final version of the manuscript.

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407

408 **Figure Legends**

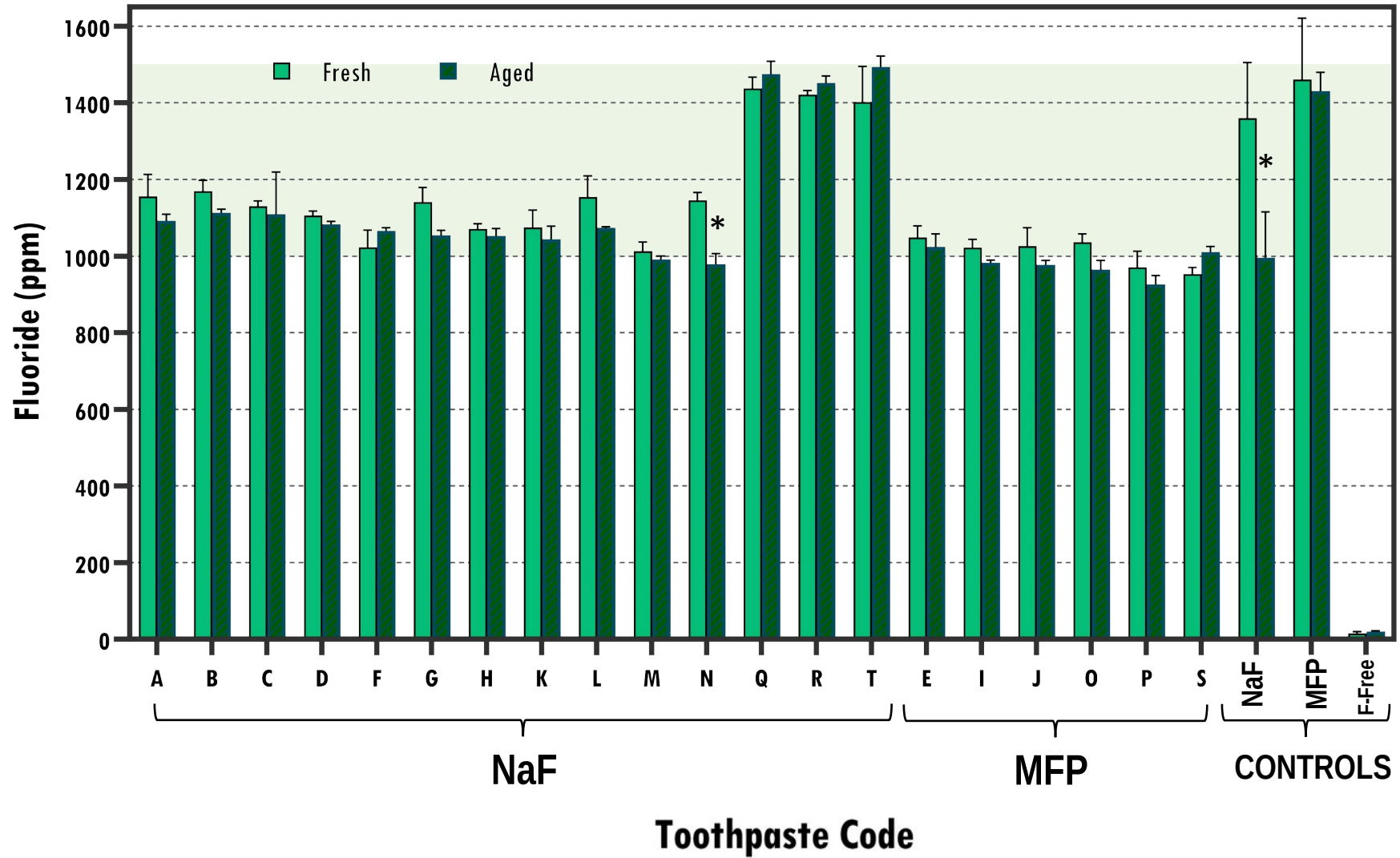
409

410 **Figure 1:** Total soluble Fluoride (TSF), in fresh and aged samples of F toothpastes based on activated charcoal with
411 NaF or MFP as a source of F. Bars corresponds to the mean, and error bars to the standard deviation (SD) (n=4). An
412 asterisk above the bars represents a statistically significant difference between fresh and aged conditions (t-test;
413 $p < 0.05$). The green demarcated area highlights the threshold of anticaries effectiveness (1,000-1,500 ppm F).

414

415 **Figure 2.** pH measurements on fluoridated charcoal-containing toothpastes. The square corresponds to the mean,
416 and error bars to the standard deviation (SD) (n=4).

417



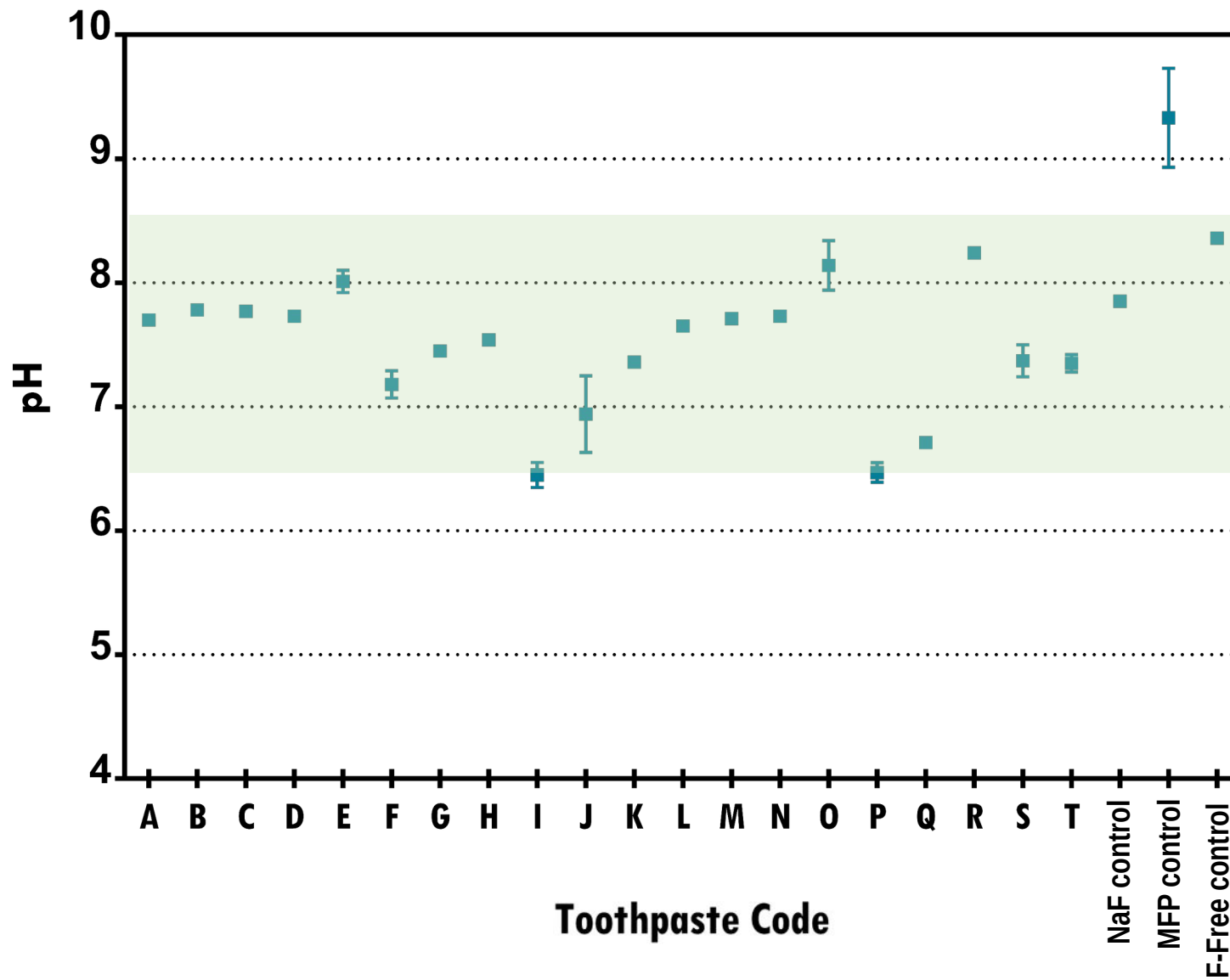


Table 1. Codes and composition of the analyzed fluoridated charcoal-containing toothpastes as declared by the manufacturer. NaF : sodium fluoride; MFP : monofluorophosphate. – NI: Not informed.

Year of Purchase and Aging	Code	Declared F (ppm)	F agent	Abrasive System	Manufacturer Country	Purchase Country	Lot #	Expiration Date
2020 - Natural Aging	A	1100	NaF	hydrated silica	NI	USA	LOT0002GR	NOV. 2021
							LOT0024GR	DEC.2021
	B	1100	NaF	hydrated silica	NI	USA	LOT9351GR	NOV.2022
							LOT9365GR	NOV.2022
	C	1100	NaF	hydrated silica	NI	USA	LOT9322GR	OCT.2022
							LOT9365GR	NOV.2022
	D	1100	NaF	hydrated silica	NI	USA	LOT00012GC	DEC.2022
							LOT9358GC	NOV.2022
	E	1000	MFP	hydrated silica	Mexico	USA	9243MX111E	AUG.2021
							8313MX112E	NOV.2020
	F	1100	NaF	hydrated silica	USA	USA	(L) 863235	APR.2021
							(L) 863236	SEP.2021
	G	1100	NaF	hydrated silica	NI	USA	LOT9189M107	JUN.2021
							LOT9182M107	MAY.2021
	H	1100	NaF	hydrated silica	NI	USA	9248UST11 D	SEP.2021
							9108UST11 C	APR.2021
	I	1000	MFP	hydrated silica	USA	USA	(L) 918222	AUG.2021
							(L) 918221	AUG.2021
	J	1000	MFP	hydrated silica	USA	USA	(L) 1779A	JUN.2021
							(L) 1399A	MAY.2021
K	1100	NaF	hydrated silica	USA	USA	LOT9319M107	OCT.2021	
L	1100	NaF		USA	Chile	L9101GR	FEB.2021	
M	1000	NaF	hydrated silica	China	Chile	9265CN123N	SEP. 2022	
						9287CN121N	OCT.2022	
N	1100	NaF	hydrated silica	USA	Chile	L9272GR	AUG.2021	
						L9228GR	JUN.2021	
O	1000	MFP	hydrated silica	México	Chile	9344MX111H	DIC.2021	
						0014MX1113H	JAN.2022	
P	950	MFP	hydrated silica	Suiza	Chile	73320633	-	
						73320644	-	
MFP Control	1450	MFP	CaCO3/silica	China	Chile	L20147T1L7ID	FEB.2023	
NaF Control	1450	NaF	hydrated silica	Mexico	Chile	0188MX1163	JUL.2022	
2023*- Artificial Aging	Q	1450	NaF	hydrated silica	Francia	Chile	20963CC	APR.2025
							20143CB	JAN.2025
	R	1450	NaF	hydrated silica	Mexico	Chile	2283MX1113	OCT.2024
							2284MX112H	OCT.2024
	S	1000	MFP	hydrated silica	USA	Chile	1278US561C	OCT.2023
							2120US561E	APR.2024
	T	1450	NaF	hydrated silica	Brazil	Chile	3025BR121D	JAN.2025
							2295BR121D	OCT.2024
	NaF Control	1450	NaF	hydrated silica	Mexico	Chile	2045MX1122	FEB.2024
							2331MX111H	NOV.2024
MFP Control	1450	MFP	CaCO3/hydrated silica	China	Chile	20250714X1	JUL.2025	
						20250617X1	JUN.2025	
F-free Control	0	-	hydrated silica	USA	Chile	1280US561C	OCT. 2023	
						1337US561C	DEC.2023	

Footnote: NaF = sodium fluoride; MFP = monofluorophosphate; CaCO3 = calcium carbonate; NI = not informed *Toothpastes did not exist 3 years ago (in 2020). They underwent an accelerated aging process in the laboratory to also assess their stability.

Condition	Code	Total F		Total Soluble F	Soluble F		% Insoluble F
		Expected	Measured		As ion		
					F ⁻	FPO ₃ ²⁻	
Fresh	A	1100	1173 ± 45.6	1156.4 ± 57.4	1171.1 ± 33.6		1.41
	B	1100	1166.7 ± 43.7	1170.2 ± 28.7	1216.9 ± 86.8		0.00
	C	1100	1108.6 ± 15.3	1129.9 ± 14.6	1160.8 ± 11.1		0.00
	D	1100	1098.4 ± 13.7	1106.2 ± 12.7	1124.3 ± 30.2		0.00
	E*	1000	1019.3 ± 37.6	1048.8 ± 30	65.7 ± 7	983.2 ± 24.8	0.00
	F	1100	1037.2 ± 25.7	1023.1 ± 45.6	1060.8 ± 36.3		1.36
	G	1100	1153.6 ± 10.5	1141.3 ± 38.4	1155.1 ± 30.2		1.07
	H	1100	1075.3 ± 8.5	1071.0 ± 13.8	1093.1 ± 26		0.00
	I*	1000	1008.0 ± 58.9	1021.8 ± 22.5	30.3 ± 2.8	991.5 ± 22.2	0.00
	J*	1000	986.4 ± 44.7	1025.6 ± 48.9	33.9 ± 1.9	991.7 ± 48.2	0.00
	K	1100	1124.1 ± 34.6	1075.2 ± 45	1130.2 ± 55.1		4.35
	L	1100	1161.2 ± 88.7	1153.8 ± 55.9	1165.6 ± 68.9		0.00
	M	1000	1033.6 ± 16.7	1012.8 ± 24.7	1071.4 ± 3.6		2.01
	N	1100	1131.2 ± 3	1145.7 ± 20.7	1142.2 ± 5.6		0.00
	O*	1000	994.1 ± 32.1	1036.3 ± 22.4	50.3 ± 4.5	986 ± 25.9	0.00
	P*	950	1041.3 ± 15.4	970.5 ± 42	316.1 ± 26.8	654.4 ± 68.2	6.80
	Q	1450	1428.6 ± 18.5	1438.1 ± 29.9	1456.7 ± 23.3		0.00
R	1450	1471.1 ± 16.1	1421.0 ± 11.2	1485.4 ± 4.9		3.40	
S*	1000	955.5 ± 7.3	952.6 ± 17.2	34.2 ± 0.4	918.4 ± 24.3	0.00	
T	1450	1436.9 ± 0.4	1402.2 ± 98.3	1388.5 ± 51.4		2.41	
NaF Control	1450	1475.0 ± 107.1	1460.9 ± 159.5	1520.8 ± 92.4		0.96	
MFP Control	1450	1456.4 ± 39.3	1359.7 ± 144.7	227.0 ± 58.8	1132.7 ± 190.7	6.64	
Non Fluoride	0	18.3 ± 2.2	15.5 ± 4.4	18.4 ± 1.4		0.00	
Aged	A	1100	1086.3 ± 28.2	1092.0 ± 16.8	1099.0 ± 9.9		0.00
	B	1100	1095.0 ± 12.4	1112.8 ± 9.9	1118.8 ± 19.6		0.00
	C	1100	1091.0 ± 113.5	1109.4 ± 109.8	1097.3 ± 104.2		0.00
	D	1100	1055.4 ± 8.8	1082.9 ± 8	1070.2 ± 9.5		0.00
	E*	1000	982.7 ± 32.9	1023.5 ± 34.1	86.0 ± 7.9	937.5 ± 26.3	0.00
	F	1100	1065.9 ± 12.7	1065.8 ± 8.6	1059.0 ± 9.4		0.003
	G	1100	1058.8 ± 9.7	1054.3 ± 12.8	1090.1 ± 25.5		0.43
	H	1100	1052.1 ± 13.6	1052.2 ± 19.5	1076.1 ± 16.8		0.00
	I*	1000	964.6 ± 5.5	982.3 ± 7.5	42.9 ± 1.2	939.4 ± 7.8	0.00
	J*	1000	958.1 ± 11	976.8 ± 12.2	45.0 ± 0.7	931.8 ± 12.1	0.00
	K	1100	1071.9 ± 17.1	1043.9 ± 34.3	1058.8 ± 1.2		2.61
	L	1100	1085.1 ± 12.8	1073.5 ± 2.9	1075.8 ± 0.4		1.07
	M	1000	976.5 ± 4.1	991.3 ± 9.2	1006.4 ± 18.3		0.00
	N	1100	995.8 ± 19.5	978.9 ± 27.6	1029.8 ± 23.4		1.70
	O*	1000	952.2 ± 15.5	964.8 ± 23.7	66.0 ± 3.4	898.8 ± 25.7	0.00
	P*	950	931.7 ± 19.3	925.7 ± 23.5	511.9 ± 16.5	413.8 ± 38.3	0.64
	Q	1450	1415.9 ± 28.6	1475.1 ± 33.0	1438.9 ± 22.4		0.00
R	1450	1448.5 ± 14.8	1451.5 ± 19.0	1460.9 ± 24.0		0.00	
S*	1000	928.4 ± 11.9	1010.8 ± 14.3	109.6 ± 5.8	900.4 ± 10.05	0.00	
T	1450	1371.9 ± 21.8	1493.7 ± 28.6	1410.7 ± 12.7		0.00	
NaF Control	1450	1443.0 ± 92.6	1431.0 ± 48.9	1444.9 ± 7.5		0.00	
MFP Control	1450	1406.2 ± 25.7	995.5 ± 119.7	314.0 ± 108.7	681.5 ± 192.8	29.21	
Non Fluoride	0	21.3 ± 4.3	20.1 ± 2.1	17.3 ± 1.1		0.00	

Footnote: NaF = sodium fluoride; MFP = monofluorophosphate , FPO₃²⁻= Monofluorophosphate ion
 *formulations with MFP and hydrated silica. NaF and MFP control values are an average between toothpastes acquired in 2020 (natural aging) and 2023 (accelerated aging). Data were not different for controls between both aging processes.

Table 2. Concentrations (ppm F or mg F/kg) of total fluoride (TF) expected and measured, total soluble fluoride (TSF) and ionic fluoride as F ion (F⁻) and monofluorophosphate ion (FPO₃²⁻) in FRESH and AGED Toothpastes (Average±standard deviation; n=4)