ABRASIVITY ASSESSMENT OF PARTICLES USED FOR “TOOTHPASTE TABLETS”

Final report

Sandra Sarembe, Carolin Ufer, Andreas Kiesow

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Sandra Sarembe

Digital unterschrieben von Sandra Sarembe
Datum: 2021.10.27 16:58:12 +02'00'

Dr. Sandra Sarembe

p.p. Maria Morawietz

Digital unterschrieben von Maria Morawietz
Datum: 2021.10.28 09:54:28 +02'00'

Andreas Kiesow

Objectives

The aim of this study was to investigate the relative dentin abrasion (RDA) of particles, which will be used for toothpaste tablets from Münt Healthcare. The assessment of the abrasion on dentin was performed according an in-house protocol which is based on ISO 11609: 2017: Dentistry - Dentifrices - Requirements, test methods and marking. This method has been established to be equivalent to the radiotracer method and will be referred to as RDA-PE – Relative Dentin Abrasion – Profilometry Equivalent.

Since two different reference particles (ISO silica used in Europe and calcium pyrophosphate used in USA) are defined in the ISO, both reference particles were used to calculate the RDA-PE of the toothpaste tablets particles.

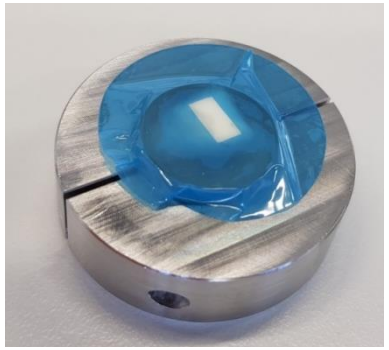
[1] ISO 11609: 2017: Dentistry - Dentifrices - Requirements, test methods and marking

Material and methods

Sample preparation

- Dentin specimens were prepared from sound human teeth by embedding in epoxy resin (EpoFix, Struers) and grinding to 4000 grit.
- Prior to the abrasion test, each specimen was taped with an adhesive tape (PRO067-7, Argon Masking Inc., Monrovia, CA, USA) to expose a defined dentin area (3 x 6 mm^2).
- All samples fulfil following requirements (according to ISO 11609, Part B):
  - Mean roughness value Ra < 0.1 µm (assessed by profilometry)
  - Embedded blocks level (plano-parallel) within 100 µm when measured side to side (assessed by profilometry)
  - Initial Vickers surface hardness (HV) of 30–70 (assessed by microhardness measurements)

Prepared dentin sample



Taped dentin sample

Material and methods

Test groups

Test products

1. Raw particles of the Peppermint toothpaste tablets with nHA (type 5)
2. Raw particles of the Tea Tree toothpaste tablets with nHA and Activated Charcoal (type 4)
3. Raw particles of the Spearmint toothpaste tablets with nHA and Sodium Fluoride (type 4)

All solid ingredients, which will be used for preparation of the toothpaste tablets, were provided from Münt Healthcare and mixed together. Before mixing, baking soda and sodium fluoride were grinded for about three minutes using a mortar and pestle. All particles were mixed together according to a recipe provided by Münt Healthcare.

10 g of the mixed ingredients were mixed with 50 ml of a reference diluent (0.5% sodium carboxymethylcellulose, 10% glycerol). This diluent was prepared by heating 50 ml glycerol to 60°C and adding 5g sodium carboxymethylcellulose. Additional 50 ml of the heated glycerol was added after the solution has been homogenized. After 60 min stirring, 900 ml distilled water were added to the mixture. The particle suspension was left to cool to room temperature slowly overnight. Reference ISO silica and calcium pyrophosphate abrasives were prepared in the same way.

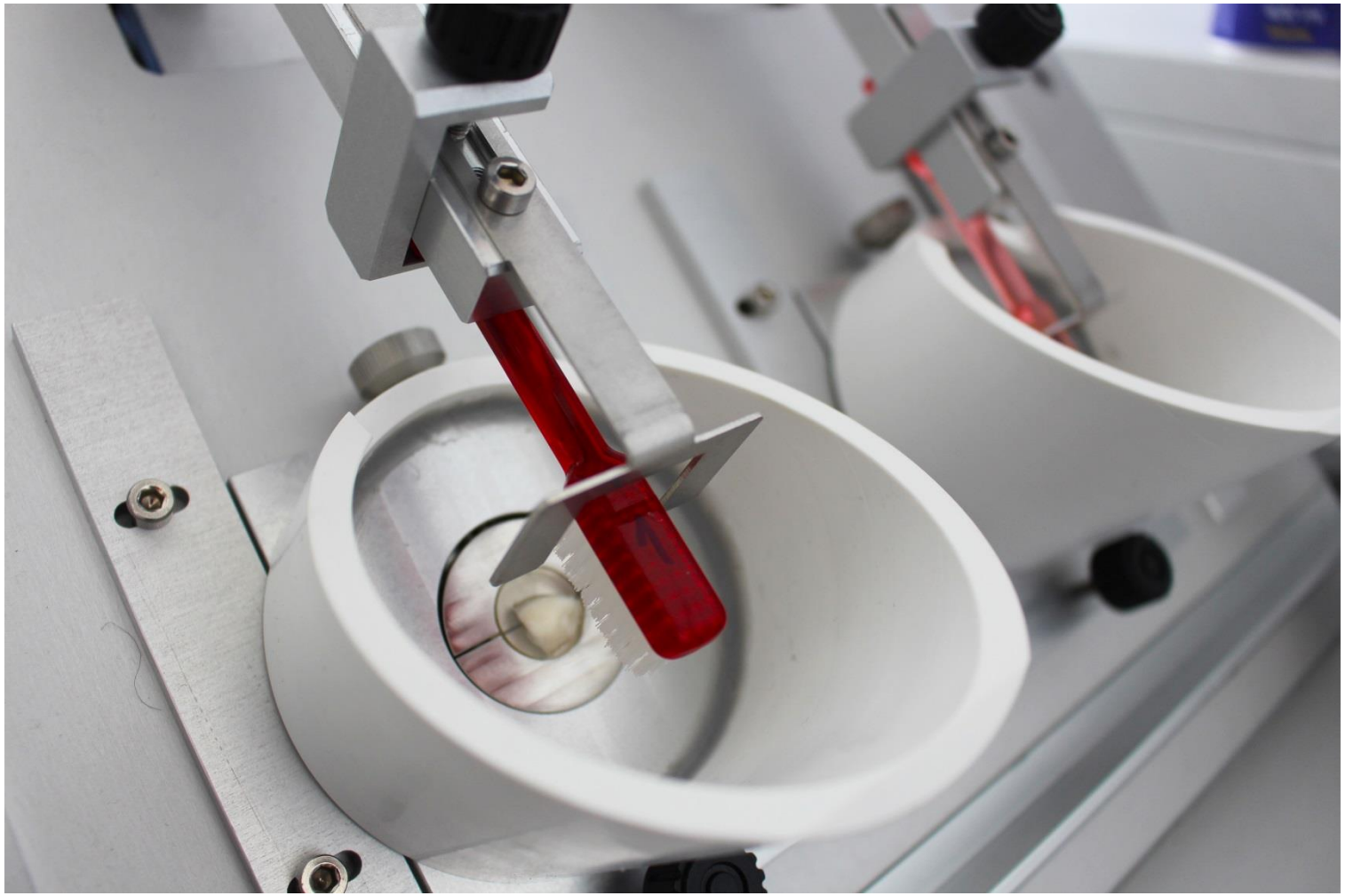
Material and methods

Sample treatment

- Prior to use, toothbrushes (Oral-B Indicator 35, medium, Procter & Gamble) were pre-conditioned for 20,000 strokes on a V8 brushing simulator (V8, JWE GmbH, Germany) in water under standard conditions. The respective brush head load was 150 g.
- Dentin samples were placed in the counter-sunk wells of the brushing simulator with a sufficient volume of toothpaste/reference slurry to ensure a coverage of least 3 mm of the slurry over the sample surface.
- The specimens were brushed for a total of 4,000 strokes, whereas one stroke is the forward and backward movement of brush heads over specimens. Respectively, after 500 strokes the samples were turned by 180 degrees.
- After brushing, the specimens were removed from the wells and washed under running tap water. The tape was removed and the abrasion depth was measured by optical profilometry (VK-X, Keyence Deutschland GmbH, Neu-Isenburg, Germany).

Material and methods

V8 brushing simulator



Material and methods

Profilometry and RDA-PE

- The relative dentin abrasion (RDA) of toothpastes is determined by a radiotracer method according to ISO 11609. Since 2017 the ISO method is supplemented of a profilometry-based method (relative dentin abrasivity – profilometry equivalent (RDA-PE)), what was applied in this study.
- Profilometric assessments of dentin wear were made by laser scanning microscopy (VK-X, Keyence Deutschland GmbH, Neu-Isenburg, Germany). Average depth of abrasion was measured as height difference of treated dentin area and reference area (covered area on the specimen surface).

Calculation of Relative Dentin Abrasivity (RDA-PE) of oral care products

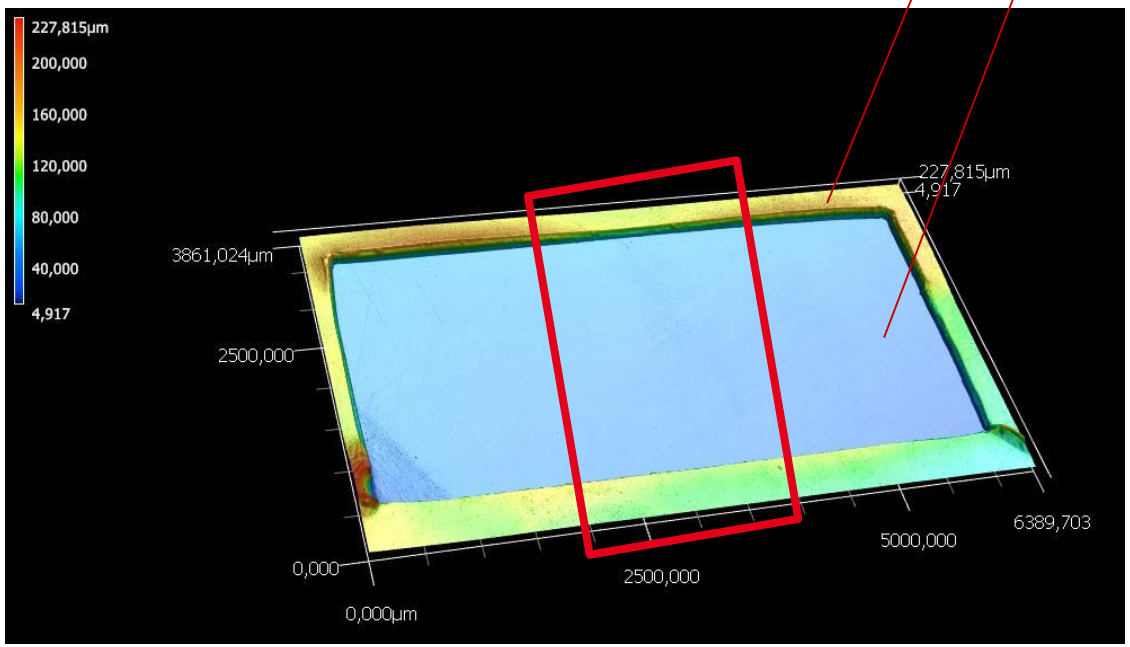
- Calculation of RDA-PE requires a proportional linearity of the reference abrasive.
- Proportional linearity for RDA-PE is the ratio of the average abrasion depth brushed for 10,000 strokes* to the abrasion depth after brushing for 4,000 strokes. This ratio shall be 2.5 and is allowed to vary between 2.2 and 2.8 (according to DIN 11609).
- On fulfilment of proportional linearity RDA-PE is calculated (after 4,000 strokes each) by:

$$\text{RDA-PE} = \frac{\textit{average abrasion depth} \text{ of dentin specimens by oral care products}}{\textit{average abrasion depth of dentin specimens by reference}} * 100$$

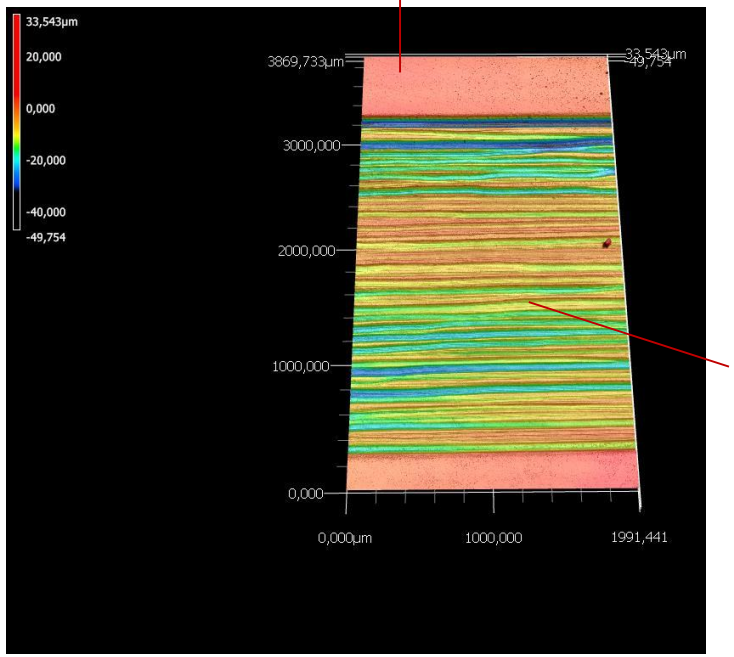
*Respectively, after 1250 cycles the samples were turned by 180 degrees.

Material and methods

Profilometry and RDA-PE



3D image of a prepared dentin sample and marked section for measuring the abrasion depth after brushing procedure



3D image of a brushed dentin sample (section)

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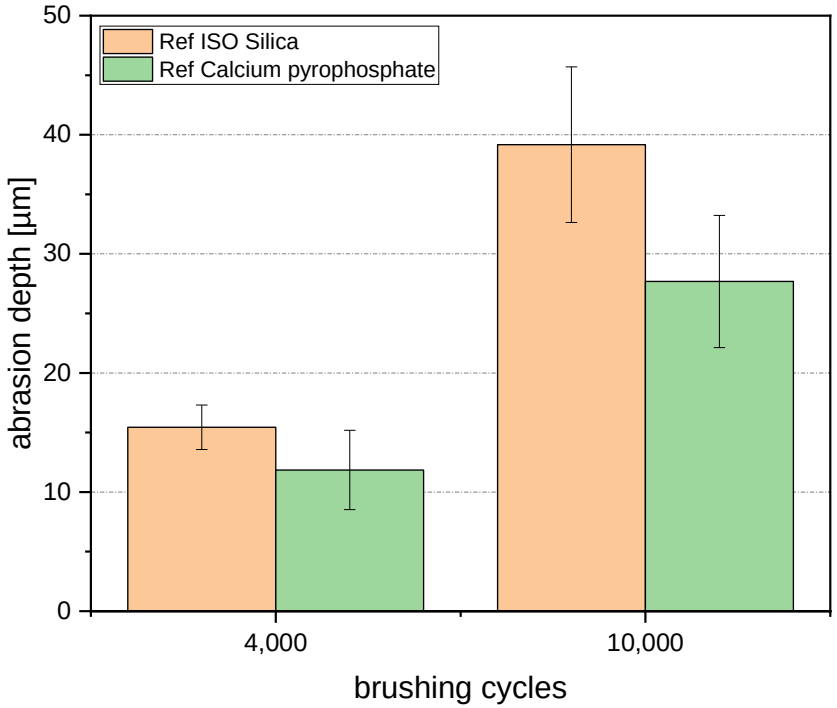
Results

Abrasion depth of the reference particles



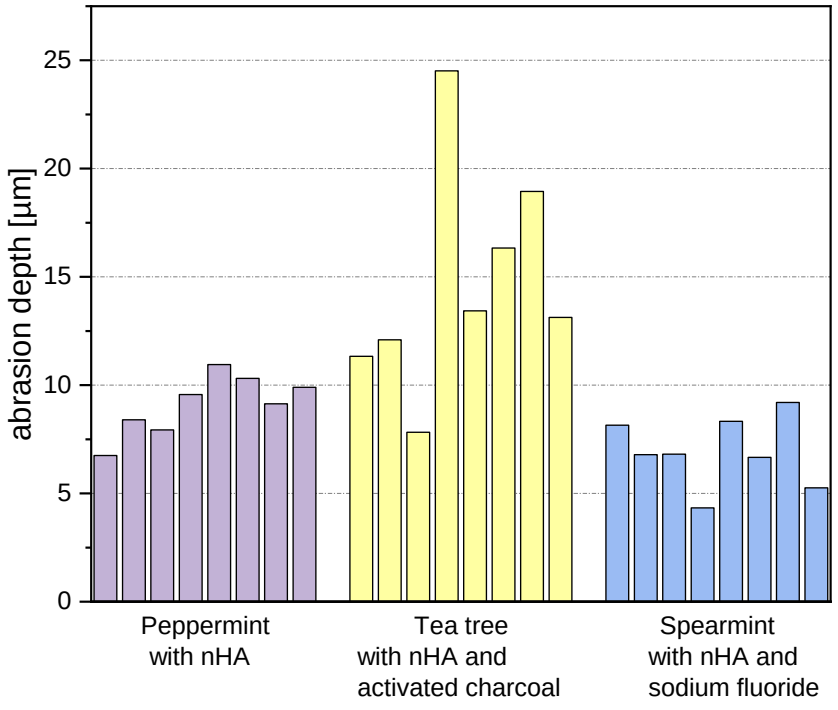
	Abrasion depth + standard deviation after brushing with ISO Silica	Abrasion depth + standard deviation after brushing with calcium pyrophosphate
4,000 cycles	15.4 ± 1.9	11.9 ± 3.3
10,000 cycles	39.2 ± 6.5	27.7 ± 5.6
	Proportional linearity fulfilled (target 2.5 ± 0.3): 39.2 / 15.4 = **2.5**	Proportional linearity fulfilled (target 2.5 ± 0.3): 27.7 / 11.9 = **2.3**

Higher abrasion depths were measured for ISO Silica than for calcium pyrophosphate. Therefore, RDA values of the test products are lower by applying ISO silica as reference than applying calcium pyrophosphate.

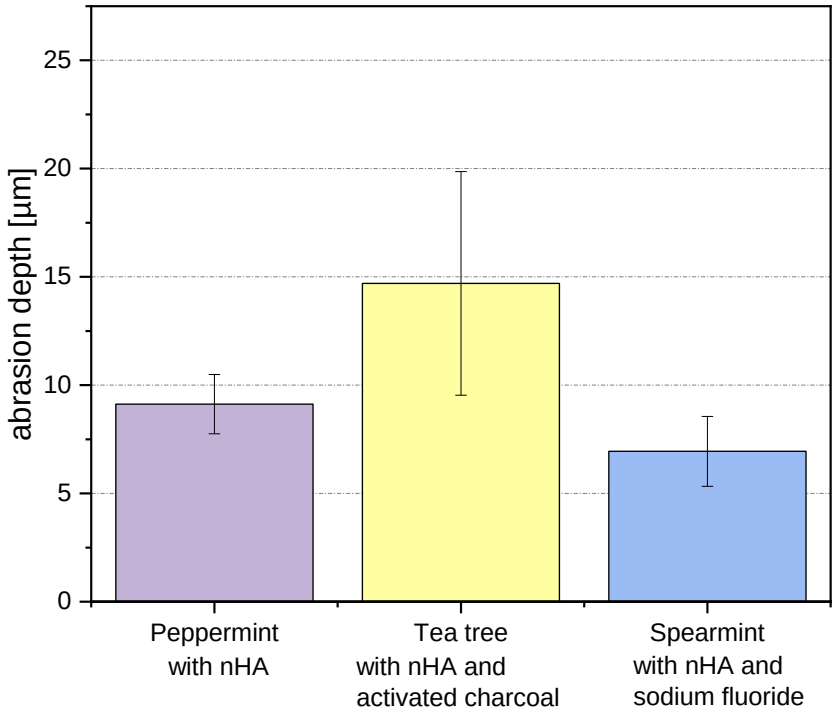
Results

Abrasion depth of the toothpaste tablets

Single values



Mean + standard deviation

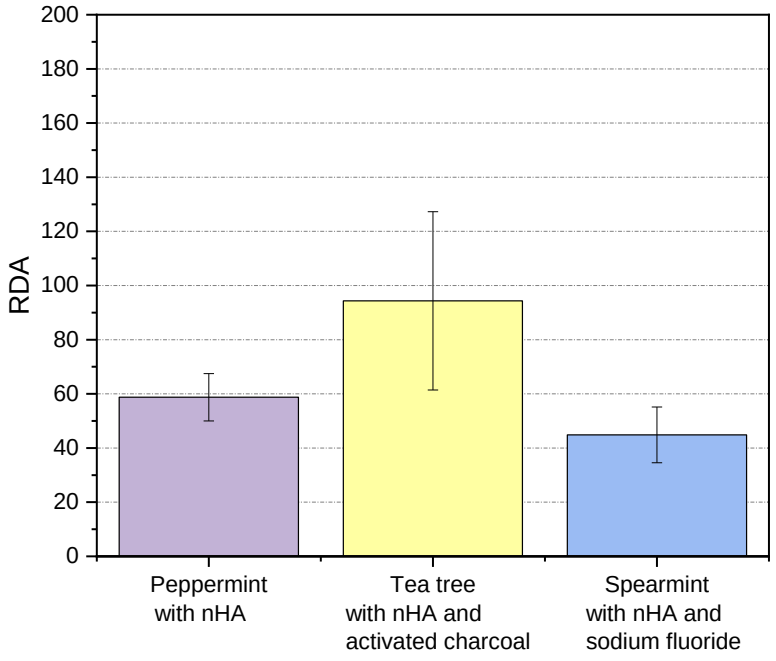


	Abrasion depth [µm] mean + standard deviation
Peppermint toothpaste tablets with nHA	9.1 ± 1.4
Tea Tree toothpaste tablets with nHA and activated charcoal	14.7 ± 5.2
Spearmint toothpaste tablets with nHA and sodium fluoride	6.9 ± 1.6

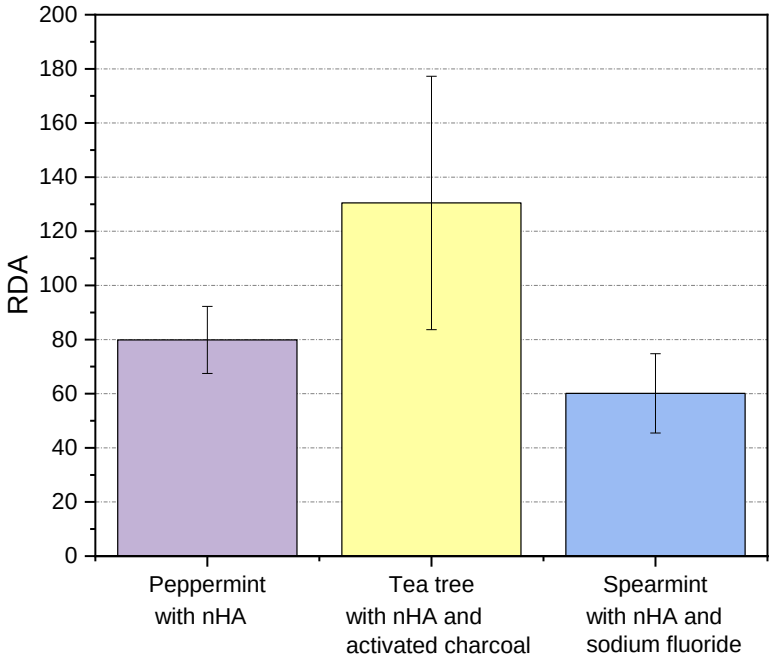
Results

RDA-PE

Mean + standard deviation based on ISO silica



Mean + standard deviation based on calcium pyrophosphate



	Mean RDA-PE + standard deviation ISO Silica based	Mean RDA-PE + standard deviation calcium pyrophosphate based
Peppermint toothpaste tablets with nHA	59 ± 8.7	80 ± 12.4
Tea Tree toothpaste tablets with nHA and activated charcoal	94 ± 32.9	130 ± 46.8
Spearmint toothpaste tablets with nHA and sodium fluoride	45 ± 10.3	60 ± 14.6

RDA-PE 200…Profilometry ISO safety limit

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Summary

- RDA-PE values of raw particles of toothpaste tablets were determined according to ISO 11609:2017 using a V8 brushing simulator. Tests were performed including the reference particles ISO silica (applied for RDA standard tests in Europe) and calcium pyrophosphate (applied for RDA standard tests in the USA).
- Based on both reference particles, medium RDA-PE values (< 100) were determined for Peppermint with nHA and Spearmint with nHA. These toothpastes tablets does not differ significantly.
- A significant higher RDA-PE value than the toothpaste tablets mentioned above was determined for Tea tree with nHA and activated charcoal.
- According to European classification, oral care products with a RDA of > 100 are classified as high abrasive, whereas according to US classification RDA of > 150 are evaluated as high abrasive.
- The RDA profilometry safety limit is currently 200 [2].

[2] Schneiderman E, Colón E, White DJ: A Profilometry-Based Dentifrice Abrasion Method for V8 Brushing Machines Part II: Comparison of RDA-PE and Radiotracer RDA Measures, J Clin Dent. 2015;26(3):61-5.

Attachment
Statistical analyses

- Statistical analyses were conducted by one-way analysis of variance (ANOVA) with post-hoc Tukey test and Brown-Forsythe test for analyses of homogeneity of variance (Origin2020, Origin Lab Corporation Company, Northampton, Massachusetts, USA). The level of significance α was set at 0.05.

ISO Silica (RDA values)	p-value	Significance 0…no significance 1…sig. differences
Tea Tree **vs** Peppermint	0.006	1
Spearmint **vs** Peppermint	0.38	0
Spearmint **vs** Tea Tree	<0.0001	1

Calcium pyrophosphate (RDA values)	p-value	Significance 0…no significance 1…sig. differences
Tea Tree **vs** Peppermint	0.006	1
Spearmint **vs** Peppermint	0.38	0
Spearmint **vs** Tea Tree	<0.0001	1