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RESEARCH ARTICLE

Evaluation of Rheological and Textural Properties of Toothpastes

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Evaluation of rheological and textural properties of toothpastes

SUMMARY

The aim of the study was to determine whether the rheological properties and texture analysis parameters of toothpastes can be evaluated together. For this purpose, ten children's toothpastes were evaluated using a rotational viscometer and texture analyzer. For each toothpaste, the shear data determined using the viscometer were plotted against shear stress data, while the shear data were plotted on a graph against viscosity data. Flow behavior index was calculated using the Ostwald de Waele equation. Following this, a texture analyzer was used to evaluate and determine each toothpastes consistency, spreadability, firmness, cohesiveness. This study showed that all of the evaluated toothpastes exhibited shear thinning. We concluded that consistency factor and the texture analyzer parameters need to be evaluated separately.

Key Words: *Toothpastes, texture analysis, viscometer, flow behavior index, shear thinning.*

Diş macunlarının reolojik ve dokusal özelliklerinin değerlendirilmesi

ÖZET

Bu çalışmanın amacı, diş macunlarının reolojik özelliklerini ve doku analiz parametrelerini birlikte değerlendirmektir. Bu amaçla piyasada bulunan on adet çocuk diş macunu üzerinde rotasyonel viskometre ve doku analiz cihazı ile birlikte değerlendirilme yapılmıştır. Her bir diş macunu için; kayma verilerine karşı viskozite verileri grafiğe geçirilmiş, kayma oranları ve kayma gerilimleri kıyaslanmıştır. Diş macunlarının akış davranış indeksi, Ostwald de Waele denklemi kullanılarak hesaplanmıştır. Ardından bir doku analizatörü ile, her bir diş macununun tutarlılığı, yayılabilirliği, sertliği, bütünlüğü belirlenmiştir. Bu çalışmada, tüm diş macunlarının kayma incelmesi özelliğini gösterdiği belirtilmiştir. Çalışmanın sonucunda; diş macunlarından hesaplanan tutarlılık faktör sonuçları ile doku analizatörü parametrelerinin ayrı ayrı değerlendirmeleri kanısına varılmıştır.

Anahtar kelimeler: *Diş macunu, doku analizi, viskometre, akış davranış indeksi, kayma incelmesi.*

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INTRODUCTION

The viscosity, flow and texture properties of a toothpaste are very important parameters that influence production and customer satisfaction. Whether a toothpaste has suitable rheological properties (viscosity, pseudoplasticity, thixotropic property, very low yield value) and texture structure depends on the properties and percentages of the substances used in its formulation. These properties and amounts are naturally different in every commercial toothpaste. A toothpaste must form a homogenous ribbon upon extrusion from the tube; must not flow out of an uncapped tube in the absence of extrusion pressure; must require minimal effort to be extruded form the tube; must form a sharp and clean ribbon of desired amount on the toothbrush following extrusion; must be able to stand up on the toothbrush, and not sink between the bristles; and must disperse quickly in the mouth after brushing is initiated. Whether a toothpaste satisfies these conditions can be determined by using suitable devices that evaluate its rheological and textural properties. Texture analysis measurements provide important information about a toothpaste's extrusion and firmness, as well as the cohesiveness and consistency of the paste mass (Laba, 1993).

The aim of this study was to determine whether the rheological properties and texture analysis parameters of toothpastes can be evaluated together. To this end, children's toothpastes marketed commercially were evaluated using a rotational viscometer and texture analyzer.

MATERIAL AND METHODS

All commercial toothpastes were given in Table 1.

Table 1. Commercial toothpastes

CODE	SERIAL	CODE	SERIAL
A1	5788463873514	A6	6642387556987
A2	5788463469871	A7	8699522267859
A3	8714789652931	A8	8699522000012
A4	6821658403415	A9	6221048405327
A5	3014260278465	A10	8690555550130

Measurement of Viscosity

In this work, in order to measure the viscosity of toothpastes, a rotational viscometer (Brookfield RVDVII, Rheocalc V2.4, cone spindle no: 52, UK.) was employed. All toothpastes were kept under same temperature. Measurements were performed constantly at 25°C. In order to distinguish the flow behavior, all tests were repeated at the shear rate of 0.02 – 0.2 for each sample. Experimental data was automatically recorded via a computer connected to a viscometer through Rheocalc software (Mojarrad *et al.*, 2014). Each experiment was carried out three times. The flow curves without intercept could be described by power low rheological model of Ostwald de Waele: Here; K (Pa.s) is the consistency index, characterizing the consistency of the system, and n is the dimensionless flow index, characterizing the degree of non-newtonian behaviour.

Texture Profile Analysis

The mechanical properties of toothpastes were determined using a software-controlled penetrometer, TA. XT Plus texture analyzer (Stable Micro Systems, UK). Each toothpaste was transferred into universal bottle (25 ml) to a fixed height of 8 cm. The Perspex probe of 10 mm diameter was twice compressed into each toothpaste at a defined rate of 2 mm. to a depth of 15 mm. Data collection and calclation were performed using the texture exponent software of instrument. Mechanical parameters such as cohesiveness, firmness (Figure 1B), adhesiveness, work of shear (Figure 1C) of the systems were calculated (Jones and Woolfson, 1997).

Hardness is defined as the force required to attain a given deformation or as the maximum peak force during the first compression cycle. Adhesiveness is defined as the negative force area for the first compression cycle and represents the work required to overcome the attractive forces between the surface of the toothpaste and the surface of the probe (Jones *et al.*, 1999). Cohesiveness (Figure 1A) defines the ratio of the area under the force-time curve produced on the second compression cycle to that produced on the second compression cycle to that produced on the first compression cycle, where successive compressions are separated by a defined recovery period. Each experiment was carried out three times.

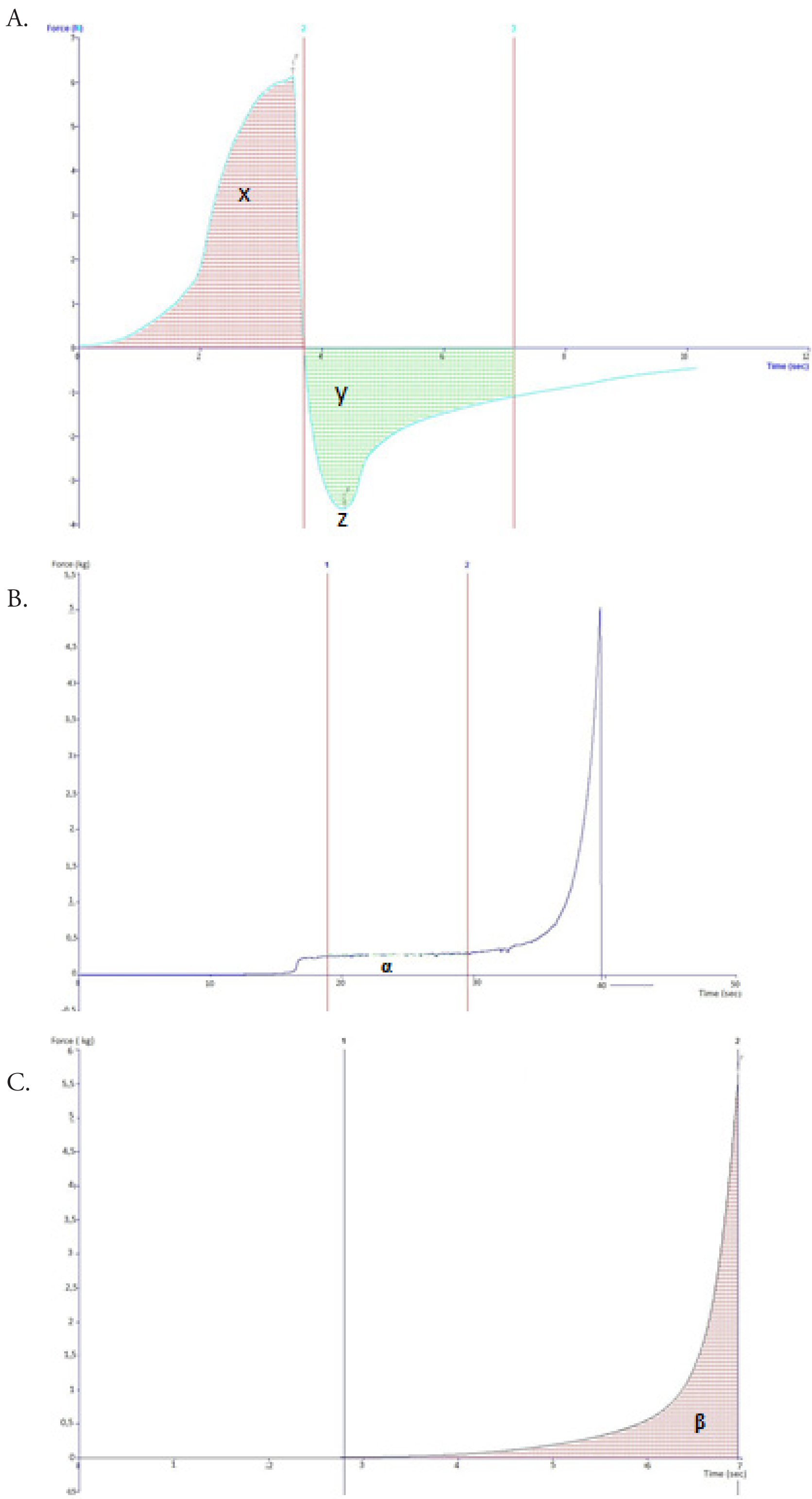

Figure 1. A. Back extrusion (x: consistency, y: index of viscosity, z: cohesiveness.), B. Forward extrusion (α: firmness.), C. Spreadability (β: work of shear).

Statistical Evaluation

The result of the texture profile analysis and viscometer analysis were statistically evaluated using one-way analysis of variance (ANOVA). $p<0.05$ was considered to be indicative of significance.

RESULT AND DISCUSSION

Rheological Behavior of Toothpastes

Figure 2 displays the viscosity (Pa.s) versus shear rate (1/sec) for different toothpastes. The result illustrated that, all the tested toothpastes exhibited a classical shear thinning behavior (n<1) and no yield stress was observed (Table 2) and K values stayed constant over time is said to be pseudoplastic system.

The flow index values (n) show that the toothpastes behaved like a shear-thinning material (with n smaller than unity).

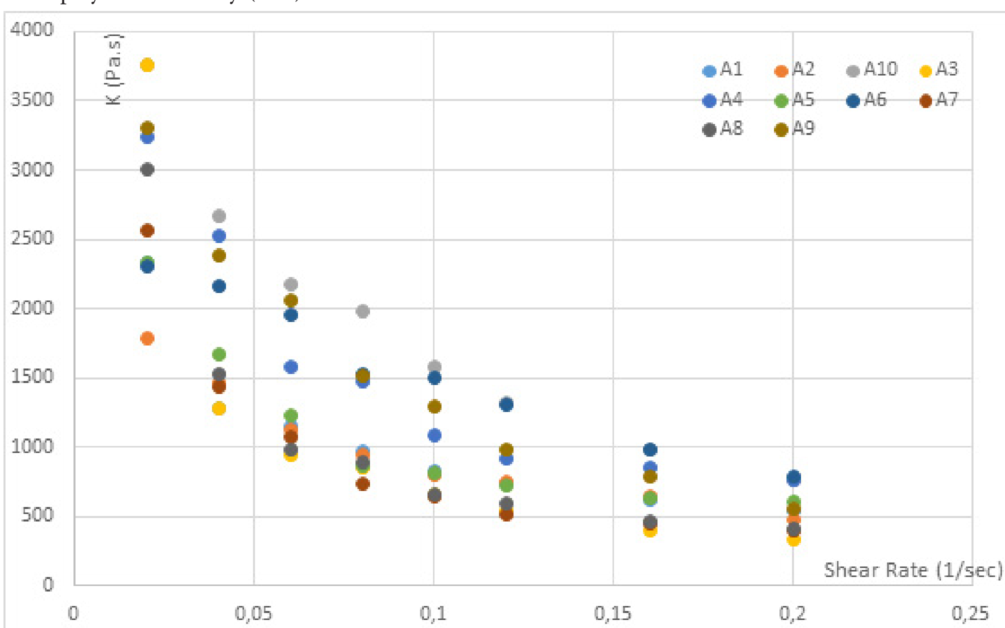

Figure 2. Viscosity versus shear rate for all toothpastes.

Table 2. Viscosity and shear thinning behavior of all toothpastes.

code	K (Pa.s)	n	r^2	code	K (Pa.s)	n	r^2
A1	208,2 ± 1,1	0,4 ± 0,01	0,999	A6	569 ± 1,2	0,61 ± 0,1	0,879
A2	225 ± 1,12	0,44 ± 0,04	0,979	A7	99 ± 1,1	0,17 ± 0,03	0,999
A3	67,3 ± 1,2	0,02 ± 0,1	0,979	A8	98 ± 1,1	0,14 ± 0,03	0,999
A4	247 ± 1,1	0,3 ± 0,1	0,979	A9	201 ± 1,2	0,24 ± 0,1	0,959
A5	203 ± 1,1	0,4 ± 0,04	0,989	A10	308 ± 1,2	0,33 ± 0,1	0,959

Mechanical Properties

The hardness introduces the necessary force to provide the deformation of toothpastes. This parameter expresses the applicability of the toothpaste to the teeth. All toothpastes should have high hardness value not to fall down from the brush (Cevher *et al.*, 2008; Gilbert *et al.*, 2013).

Textural analysis showed significant variations indicating a great dispersion of the textural properties among all toothpastes. The textural data and rheological values (K) attempt to link together. Correlation coefficients were calculated to analyze correlation between both sets (Figure 2-7). Unfortunately, the textural properties were not correlated (except for work of shear) to the K values.

A linear regression analyses with the textural properties revealed a significant correlation. These values are related to each other: the more the firmness, the more the work of shear is. The more the consistency, the more the cohesiveness is. The more the firmness, the more the consistency is. The hardness values of the toothpastes increased due to the increase in the consistency of toothpastes (Table 3).

Table 3. Mechanical properties of all toothpastes (n = 3).

CODE	BACK EXTRUSION			FORWARD EXTRUSION	SPREADABILITY
	CONSISTENCY	COHESIVENESS	IND. OF VISC	FIRMNESS	WORK OF SHEAR
	(gsec area)	(g force)	(gsec area)	(g force)	(gsec area)
A1	2422	413,3	1747,3	324	2125,1
A2	892,3	307	886	356	2239,4
A3	4217,3	433	2344	278,2	2279,2
A4	3458,3	679,2	2417,3	692	2722
A5	6593	906	5366	678,3	2721,4
A6	806,3	346	635,3	496	2442
A7	1014	357	868,4	352	2039,4
A8	2576	521,1	1652	404	2125,1
A9	9875	887,3	6490	859,1	2973,4
A10	444	353	580,2	492	2417

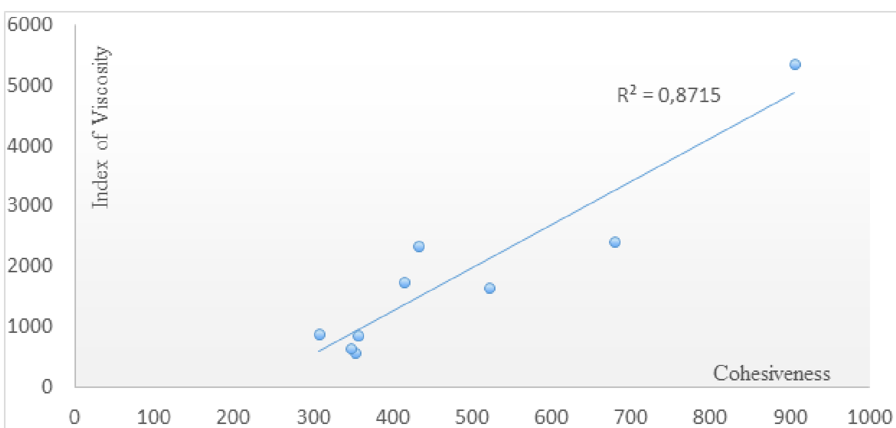

Figure 3. Index of viscosity versus cohesiveness for all toothpastes.

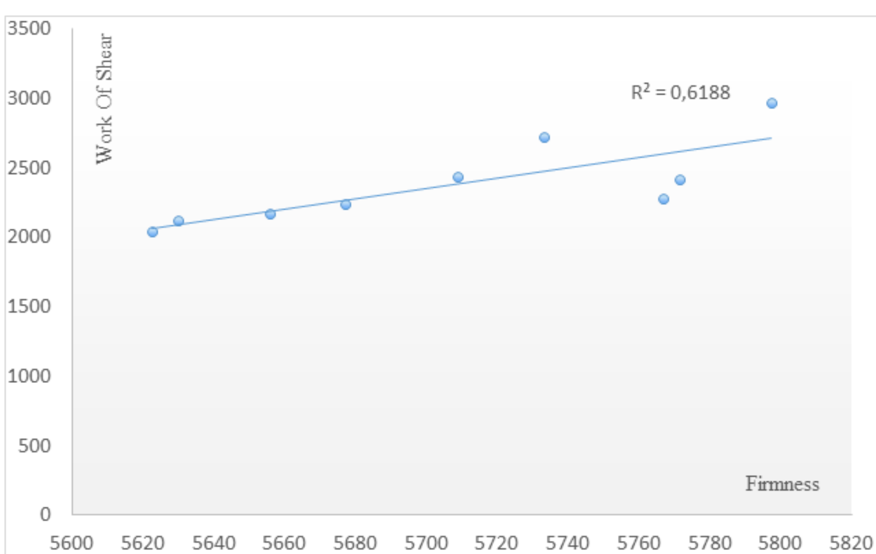

Figure 4. Work of shear versus Firmness for all toothpastes.

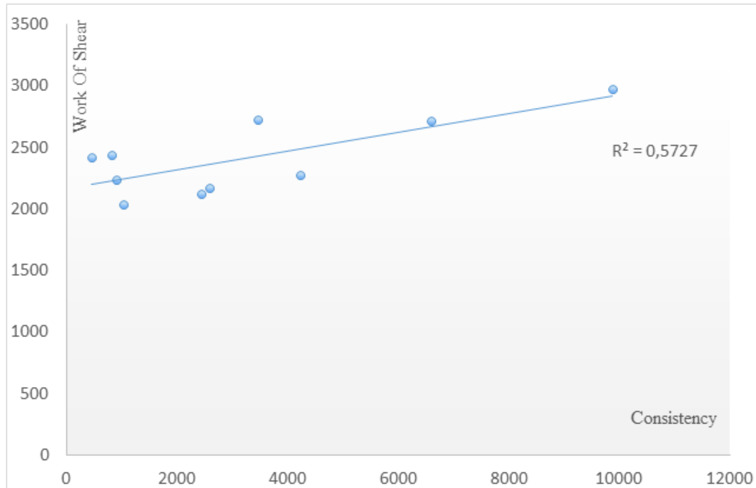

Figure 5. Work of shear versus Consistency for all toothpastes.

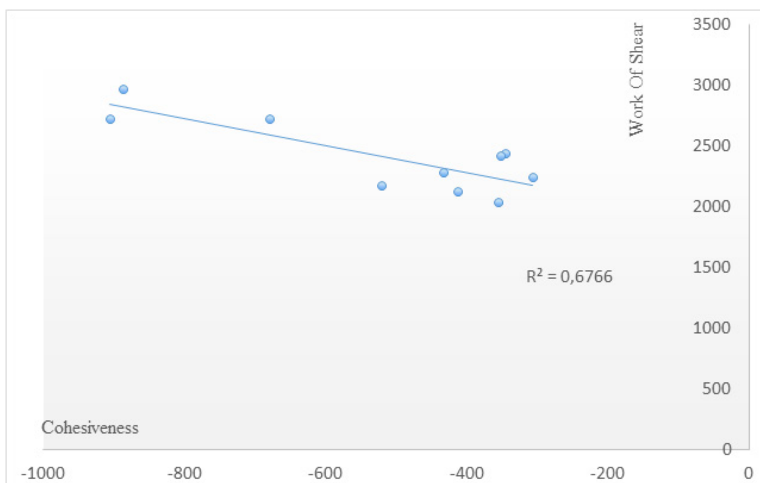

Figure 6. Work of shear versus Cohesiveness for all toothpastes.

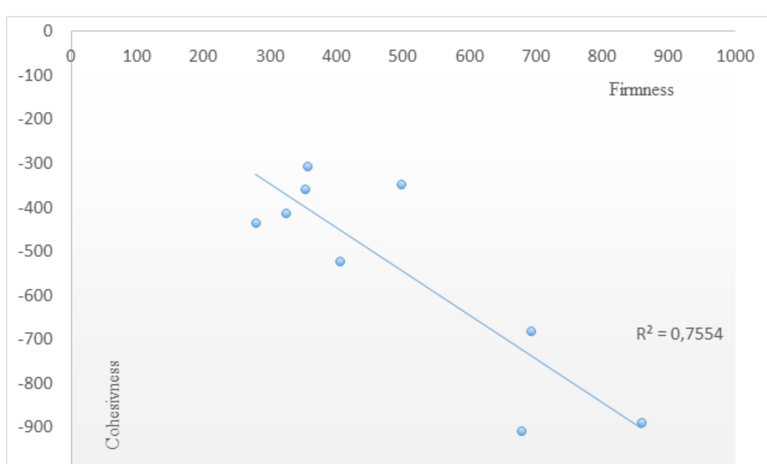

Figure 7. Firmness versus Cohesiveness for all toothpastes.

CONCLUSION

Textural analysis provides direct information on cohesiveness, consistency, firmness and hardness of toothpastes. Firmness and work of shear express the applicability of the toothpaste to the toothbrush; cohesiveness, consistency and index of viscosity can be an indicator for the retention time on the toothbrush and can describe spatial aspects of structural reformation following paste compression and early removing from container. Spreadability indicates minimal effort to be extruded from the tube. For some reasons, the textural analysis can be used to characterize products for *in vitro* quality control together with the viscometric measurement. The more firmness, the more the consistency, cohesiveness and work of shear are. Among all toothpastes, A9 has the most suitable product for its mechanical properties.

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