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THE QUALITY OF KETCHUPS FROM THE CZECH REPUBLIC'S MARKET IN TERMS OF THEIR PHYSICO-CHEMICAL PROPERTIES

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ABSTRACT
Ketchup is a tomato-based condiment with a tang contributed by vinegar, sugar, salt and spices. Physical and chemical quality requirements for ketchup are regulated in the Czech Republic by Decree No. 157/2003 as amended. The main monitored parameters determining the quality of ketchups are total tomato content, total soluble solids, total organic acids and total salt content. In this work the following parameters were monitored in a total of eight ketchups from the commercial markets in the Czech Republic: pH, total solids, total soluble solids, citric acid content, acetic acid content, lycopene content, fructose, glucose and sucrose content and content of Ca, K, Mg and Na. In addition to chemical analyses, rheological measurements were performed and dynamic viscosity and yield stress were determined. The results obtained were statistically processed and the hypothesis i) whether the sales price of ketchups is related to the quality of ketchups expressed in chemical composition and ii) whether the chemical composition affects the rheological properties of ketchups has been verified. The Pearson correlation matrix showed very good correlation between the total solids and tomato content in the ketchup (R = 0.8464) as well as between the total soluble solids and tomato content in the ketchup (R = 0.8583). Another significant correlation was found between total soluble solids and total saccharides content in ketchup (R = 0.7309) as well as between potassium content and and tomato content in the ketchup (R = 0.8864). The chemical composition of ketchups did not significantly affect the dynamic viscosity of ketchups, however strong correlation between tomato content in ketchup and between yield stresses was found (R = 0.8436). No correlation was found between the ketchup price and chemical composition of ketchup, however cheaper ketchups contained more salt.

Keywords: tomatoes; ketchup; PCA; chemical analysis; rheology

INTRODUCTION
Vegetables are an essential part of rational human nutrition. The world's most cultivated vegetables include tomatoes that are consumed mainly fresh, but they are also used for production of tomato juice or puree, which is the main raw material for the production of ketchup **(Burton-Freeman and Reimers, 2011)**. Ketchup is one of the most common flavouring agents. In addition to essential nutrients, saccharides and fibre it contains significant amounts of vitamin C, lycopene and other nutritionally important substances **(Canene-Adams et al., 2005)**.

Ketchup means roughly two to four times thickened tomato puree. The taste of ketchup is adjusted with salt, vinegar, sweetener and spice extracts. The stabilization of the resulting product requires the stabilizers (most often modified starches in an amount of about 2 - 5%) to prevent the distribution of the solid and liquid content and simultaneously to modify the consistency of the ketchup, which is to be smooth and glossy **(Hayes et al., 1998)**. Physical and chemical quality requirements for ketchup are regulated in the Czech Republic by Decree No. 157/2003 as amended. This decree states that in ketchups containing at least 12% total soluble solids, determined by refractometry, the refractometric total solid content of tomato raw material must be at least 7%. For ketchups marked as Prima, Extra or Special with refractometric total solid content at least 30%, shall be at least 10% of refractometric solids introduced with tomato raw material. Other ketchup parameters to be followed are the maximum amount of salt (up to 3%) and maximum amount of total acid (2.2% expressed as acetic acid). Rheological properties of ketchups are not regulated by decree or law, however food rheology is important in quality control during food manufacture and processing. Rheological properties of ketchups helps producers to determine ingredient functionality in product development, to predict product performance and product acceptance by consumers or to test the shelf life of product (**Norton et al., 2011**).

Scientific hypothesis

Two hypotheses were tested in this study. First, whether higher product price means higher quality for consumers in connection with the composition of the product and the other, whether the chemical composition of ketchups affects their rheological behavior. In addition, it was verified that all analyzed ketchups complied with the applicable legislation in terms of physical and chemical quality requirements.

MATERIAL AND METHODOLOGY

Sample preparation

For determination of organic acids 2 g of sample was extracted with 20 mL of ultrapure water (Elga pure lab classic, Veolia water systems Ltd., UK), in a 50 mL centrifugation tube placed on vertical skake table (GFL, Germany). After 1h of extraction, samples were centrifuged at 6000 rpm in centrifuge (EBA 21, Hettich, Germany), supernatant was filtered using filter with 0.45 µm pore size (Labicom, Czech Republic) and filled up to 50 mL in a volumetric flask with an ultrapure water.

Sample for analysis of saccharides was prepared by the same way as described in the case of organic acid, with the difference that ethanol (VWR, Germany) and ultrapure water in 4:1 volume ratio was used for the extraction.

Sample for elemental analysis was prepared using wet ashing method in a microwave oven (Milestone 1200, Milestone, Italy). For decomposition of sample matrix a mixture of nitric acid (6 mL, Analytika, Czech Republic) and hydrochloric acid (2 mL, Analytika) was used. After the decomposition sample was filtered using filter with 0.45µm pore size and filled up to 25 mL in a volumetric flask with an ultrapure water.

For a lycopene determination 0.1 g of sample was weighed to a 20 mL centrifugation tube and 8 mL of mixture of hexane, ethanol and acetone (VWR) in 2:1:1 volume ratio was added to the sample. The sample was well mixed on a vortex and left to stay for 10 min in a dark place. After 10 min 1mL of ultrapure water was added to the sample and the sample was well mixed and stored for 10 min in dark place again. The upper layer of the sample was then collected for the analysis.

Chemical analysis

Total solids were determined according to EN method **(CSN EN 12145, 1997)**. The pH value was measured using pH meter with combinated electrodes (WTW, Germany). Total soluble solids were determined by table refractometer (Kruss AR4, Germany). Organic acids were determined using ion chromatography (Metroohm 850 professional IC, Metroohm, Switzerland) with conductivity detector. A Metrosep organic acids column (250/7.8 mm) was used as stationary phase and 15% acetone (VWR) in 0.5 $mmol.l^{-1}$ sulphuric acid (Analytika) was used as a mobile phase. An Agilent Infinity 1260 liquid chromatograph (Agilent Technologies, USA) equipped with ELSD detector was used for determination of saccharides. A Prevail Carbohydrates ES column (250/4.6 mm) was used as a stationary phase and acetonitrile (VWR) mixed with water in 75:25 volume ratio was used as mobile phase. Lycopen content was determined according to the method described by **Suwanaruang (2016)** using Helios gamma spectrophotometer (Spectronic Unicam, Great Britain). An elemental analysis was performed using ICP-OES (Ultima 2, Horiba Scientific, France) according to procedure described by **Diviš et al. (2015)**.

Rheological analysis

The flow properties were determined on a rheometer Discovery HR-2 (TA Instruments) using 25 mm diameter plate-plate steel geometry. The measuring temperature was 25°C, conditioning step was 2 min, measuring slit was 28 µm, shear rate range was 0.1 – 1000 s^{-1}, the number of points per decade in logarithmic mode was 6, measurement time of one point was 10s and the number of measurements per point was 3. After the sample was subjected to the basic flow test, the same sample was again subjected to a flow test with the same rheometer parameters after an 8 minute conditioning step. By this way sample relaxation was detected. From the shear stress to shear rate dependence yield stress and flow index were calculated using the Herschel-Bulkley equation in the form: $\tau = \tau_0 + K \cdot \gamma^n$, where τ is the shear stress, τ_0 is the yield stress, K is the consistency coefficient and n is the flow index.

Statisic analysis

All samples were prepared in duplicates and each sample analysis was performed three times. Before the main data analysis, results were tested for outliners and data distribution. Grubbs test for outliners did not revealed any outlined values within all analyzed parameters and data showed a normal gaussian distribution. All parameters were analyzed by Pearson correlation matrix and independent variables were further classified using principal component analysis (XL Stat, version 2015.5, Addinsoft, France). Tukey's comparative test using the 0.05 significance level has been performed to find means that are significantly different from each other.

RESULTS AND DISCUSSION

Total solids and total soluble solids are an important quality factors in the tomato processing industry **(Thakur et al. 1996)**. According to valid legislation in the Czech Republic, ketchups must contain at least 12% total solids, determined by refractometry. In the case of ketchups marked as "Prima", "Extra" and "Special" the total solids determined by refractometry must be at least 30%. Total solids content of the analyzed samples ranged from 23.6 to 31.4% **(Table 1)**. The results are in agreement with those obtained by **Lehkoživová et al. (2009)**, or **Sharoba et al. (2005)**. All ketchup samples, including samples K2 and K6 marked as "Extra", were in compliance with the requirements of the applicable legislation. There was good correlation (R = 0.8583) between the total solids content and the tomato content in the ketchup as well as between the total soluble solids and the tomato content in the ketchup (R = 0.8464) (**Table 5**). However, ketchups with a smaller amount of tomatoes, which have a relatively high total solids content, in particular samples K3 and K5, do not suit the model of correlation. For these ketchups, it is to be assumed that other vegetables (such as carrots, onions) have been used to produce them, in addition to

Table 1 Basic physicochemical parameters of ketchup samples K1-K8.

Sample	pH (1±SD)	Total solids ($mg.kg^{-1}$±SD)	TSS* (%±SD)	Tomato content ($g.100g^{-1}$)**
K1	3.98 ±0.05[a]	25.0 ±0.3[f]	27.71 ±0.05[g]	140
K2	3.94 ±0.05[ab]	31.4 ±0.2[a]	35.44 ±0.05[a]	240
K3	3.72 ±0.05[e]	26.5 ±0.2[e]	30.50 ±0.05[f]	148
K4	3.85 ±0.05[cd]	26.4 ±0.2[e]	31.55 ±0.05[c]	170
K5	3.74 ±0.05[de]	27.8 ±0.2[c]	31.33 ±0.05[d]	151
K6	3.98 ±0.05[a]	28.2 ±0.3[b]	31.06 ±0.05[e]	200
K7	3.74 ±0.05[de]	27.4 ±0.1[d]	32.17 ±0.05[b]	210
K8	3.82 ±0.05[bc]	23.6 ±0.1[g]	27.12 ±0.05[h]	140

Note: Values in the same column with different letters are significantly different at p <0.05. * TSS = total soluble solids, ** Declared content on packaging.

Table 2 Saccharide content in ketchup samples K1-K8.

Sample	Saccharides				
	fructose ($mg.g^{-1}$±SD)	glucose ($mg.g^{-1}$±SD)	sucrose ($mg.g^{-1}$±SD)	Σ Saccharides ($mg.g^{-1}$±SD)	Σ Saccharides* ($mg.g^{-1}$)
K1	47.0 ±0.3[c]	66.4 ±0.8[a]	n.d.	113.4 ±1.1[e]	240
K2	40.9 ±0.5[e]	57.6 ±0.3[c]	82.7 ±0.5[e]	181.2 ±1.3[c]	344
K3	43.7 ±0.4[d]	43.2 ±0.4[f]	115.2 ±0.7[c]	205.7 ±1.5[a]	232
K4	53.9 ±0.3[a]	49.3 ±0.3[d]	82.6 ±0.6[e]	185.8 ±1.2[b]	260
K5	24.4 ±0.2[g]	24.5 ±0.2[h]	117.0 ±0.8[b]	165.9 ±1.2[d]	250
K6	34.8 ±0.3[f]	28.9 ±0.2[g]	121.0 ±0.6[a]	184.7 ±1.1[b]	240
K7	48.1 ±0.6[b]	45.7 ±0.4[e]	89.3 ±0.4[d]	183.1 ±1.4[bc]	240
K8	46.0 ±0.3[c]	62.8 ±0.5[b]	n.d.	108.8 ±0.8[f]	276

Note: The chemical composition is on a wet weight basis. Values in the same column with different letters are significantly different at p <0.05. *Declared content on packaging.

tomatoes, which increased the total solids content. Only ketchup sample K5, however, declares on the package the use of dried vegetables (onion, garlic).

Total soluble solids are generally closely related to saccharide content. In this work a good correlation was also found between the total soluble solids and total saccharides (R = 0.7280) (**Table 5**). The main saccharides in ketchup samples were glucose and fructose. In most samples sucrose was also determined. The amount of carbohydrates is related to the tomato variety and tomato ripening used to produce ketchup. Other saccharides can be added to the ketchups during their sweetening. The concentration of fructose in ketchup samples varied from 24.4 $mg.g^{-1}$ to 53.9 $mg.g^{-1}$, while concentration of glucose varied between 24.5 $mg.g^{-1}$ and 66.4 $mg.g^{-1}$ and sucrose between non-detectable quantities and 121 $mg.g^{-1}$ (**Table 2**). Similar values were measured by **Sharoba et al. (2005).** The total amount of saccharides obtained by the sum of glucose, fructose and sucrose concentration does not agree with the data on the packaging, indicating higher sacharide content. This difference can be explained by the addition of starch or xanthan to ketchup, which affect its texture. These added polysaccharides may make up the difference between the total saccharide content and the declared total saccharide content.

The quantity of tomatoes used for ketchup production correlates fairly well with the lycopene content in the ketchups (R = 0.6704) (**Table 5**). The amount of lycopene in ketchups ranged between 0.056 and 0.266 $mg.g^{-1}$ (**Table 3**). **Wawrzyniak et al. 2005** mentioned the amount of lycopene in ketchup in the range of 0.07 – 0.140 $mg.g^{-1}$. The content of lycopene in ketchup is dependent on lycopene content in tomatoes used for tomato puree production and also on ketchup production process in which tomatoes undergo multistep mechanical and heat-related processing operations that could potentially reduce lycopene content in final product (**Mendelova et al., 2013**).

The pH value is one of the most important factors affecting the growth and biochemical activity of microorganisms in food. In the case of chemically preserved vegetables, a pH of 4.1 is required under the legislation. The pH of ketchups is affected by the natural organic acid content in tomatoes (e.g. malic or ascorbic acid) and by added preservatives. The pH of the individual samples ranged between 3.72 and 3.98 (**Table 1**). Similar values were measured by **Sharoba et al. (2005)** while **Lehkoživová et al. (2009)** measured generally higher pH, between 4.1 and 4.3. The most important acids in ketchup are acetic and citric acid. The acetic acid content is related to the technological process of ketchup production, when vinegar is added to the ketchup as a flavoring agent. Citric acid is the most commonly used pH regulator in food. The acetic acid content in analyzed samples varied from 16.8 to

Table 3 Organic acids content and lycopene content in ketchup samples K1-K8.

Sample	Citric acid ($mg.g^{-1}$±SD)	Acetic acid ($mg.g^{-1}$±SD)	Lycopene ($mg.g^{-1}$±SD)
K1	3.7 ±0.2[d]	16.8 ±0.3[f]	0.108 ±0.003[d]
K2	7.7 ±0.3[a]	31.6 ±0.3[a]	0.241 ±0.017[b]
K3	2.9 ±0.1[e]	26.4 ±0.4[c]	0.056 ±0.005[e]
K4	5.4 ±0.3[c]	29.9 ±0.5[b]	0.132 ±0.007[c]
K5	4.3 ±0.2[d]	22.1 ±0.2[d]	0.135 ±0.003[c]
K6	5.9 ±0.4[c]	22.1 ±0.3[d]	0.266 ±0.006[a]
K7	6.9 ±0.3[b]	29.6 ±0.6[b]	0.103 ±0.007[d]
K8	3.9 ±0.2[d]	19.1 ±0.5[e]	0.123 ±0.004[cd]

Note: The chemical composition is on a wet weight basis. Values in the same column with different letters are significantly different at p <0.05.

Table 4 Mineral composition of ketchup samples K1-K8.

Sample	Elemental compositon				
	Ca ($mg.g^{-1}$±SD)	K ($mg.g^{-1}$±SD)	Mg ($mg.g^{-1}$±SD)	Na ($mg.g^{-1}$±SD)	NaCl ($mg.g^{-1}$±SD)
K1	0.308 ±0.009[e]	2.483 ±0.084[e]	0.136 ±0.003[e]	6.383 ±0.138[c]	16.22 ±0.14[c]
K2	0.461 ±0.016[b]	4.979 ±0.033[a]	0.367 ±0.006[a]	5.736 ±0.089[d]	14.61 ±0.09[d]
K3	0.416 ±0.012[c]	1.759 ±0.040[g]	0.137 ±0.003[e]	4.974 ±0.026[e]	12.62 ±0.03[e]
K4	0.417 ±0.004[c]	3.771 ±0.126[c]	0.198 ±0.005[c]	7.816 ±0.161[b]	19.95 ±0.16[b]
K5	0.217 ±0.005[f]	2.103 ±0.083[f]	0.104 ±0.003[f]	4.606 ±0.036[f]	11.71 ±0.04[g]
K6	0.386 ±0.007[d]	2.899 ±0.070[d]	0.175 ±0.004[d]	3.570 ±0.044[g]	9.13 ±0.04[h]
K7	0.910 ±0.013[a]	4.394±0.074[b]	0.293 ±0.004[b]	8.865 ±0.189[a]	22.52 ±0.19[a]
K8	0.331 ±0.005[e]	2.001 ±0.032[f]	0.111 ±0.003[f]	4.708 ±0.066[ef]	12.06±0.07[f]

Note: The chemical composition is on a wet weight basis. Values in the same column with different letters are significantly different at p <0.05.

Table 5 Pearson correlation matrix.

variables	variables									
	A	B	C	D	E	F	G	H	I	J
A	1	0.8583	0.8464	0.6704	0.4997	0.8436	0.0687	0.8864	0.2268	0.1852
B	0.8583	1	0.9476	0.4831	0.7309	0.5870	0.3457	0.7689	0.2012	0.4769
C	0.8464	0.9476	1	0.6409	0.6262	0.6830	0.4164	0.6671	-0.0371	0.3214
D	0.6704	0.4831	0.6409	1	0.1382	0.7735	0.0631	0.4488	-0.3848	-0.1086
E	0.4997	0.7309	0.6262	0.1382	1	0.4286	0.2798	0.3349	0.0941	0.9019
F	0.8436	0.5870	0.6830	0.7735	0.4286	1	-0.0124	0.5673	-0.1030	0.1473
G	0.0687	0.3457	0.4164	0.0631	0.2798	-0.0124	1	-0.2001	-0.5419	0.3437
H	0.8864	0.7689	0.6671	0.4488	0.3349	0.5673	-0.2001	1	0.5920	0.0422
I	0.2268	0.2012	-0.0371	-0.3848	0.0941	-0.1030	-0.5419	0.5920	1	0.0333
J	0.1852	0.4769	0.3214	-0.1086	0.9019	0.1473	0.3437	0.0422	0.0333	1

Note: A = tomato content, B = total soluble solids, C = total solids, D = lycopene, E = Σsaccharides, F = yield stress, G = unit price, H = potassium content, I = salt, J = dynamic viscosity.

31.6 $mg.g^{-1}$ and citric acid content ranged from 2.9 to 7.7 $mg.g^{-1}$ (**Table 3**). The acetic acid content was in agreement with results obtained by **Lehkoživová et al. (2009)** and by **Porretta (1991)**, however **Porreta (1991)** measured higher concentrations of citric acid in ketchup (average value = 16.6 $mg.g^{-1}$). For sample K7 where the second highest citric acid content was measured, the manufacturer declares on the package the use of lemon concentrate for the production of ketchup.

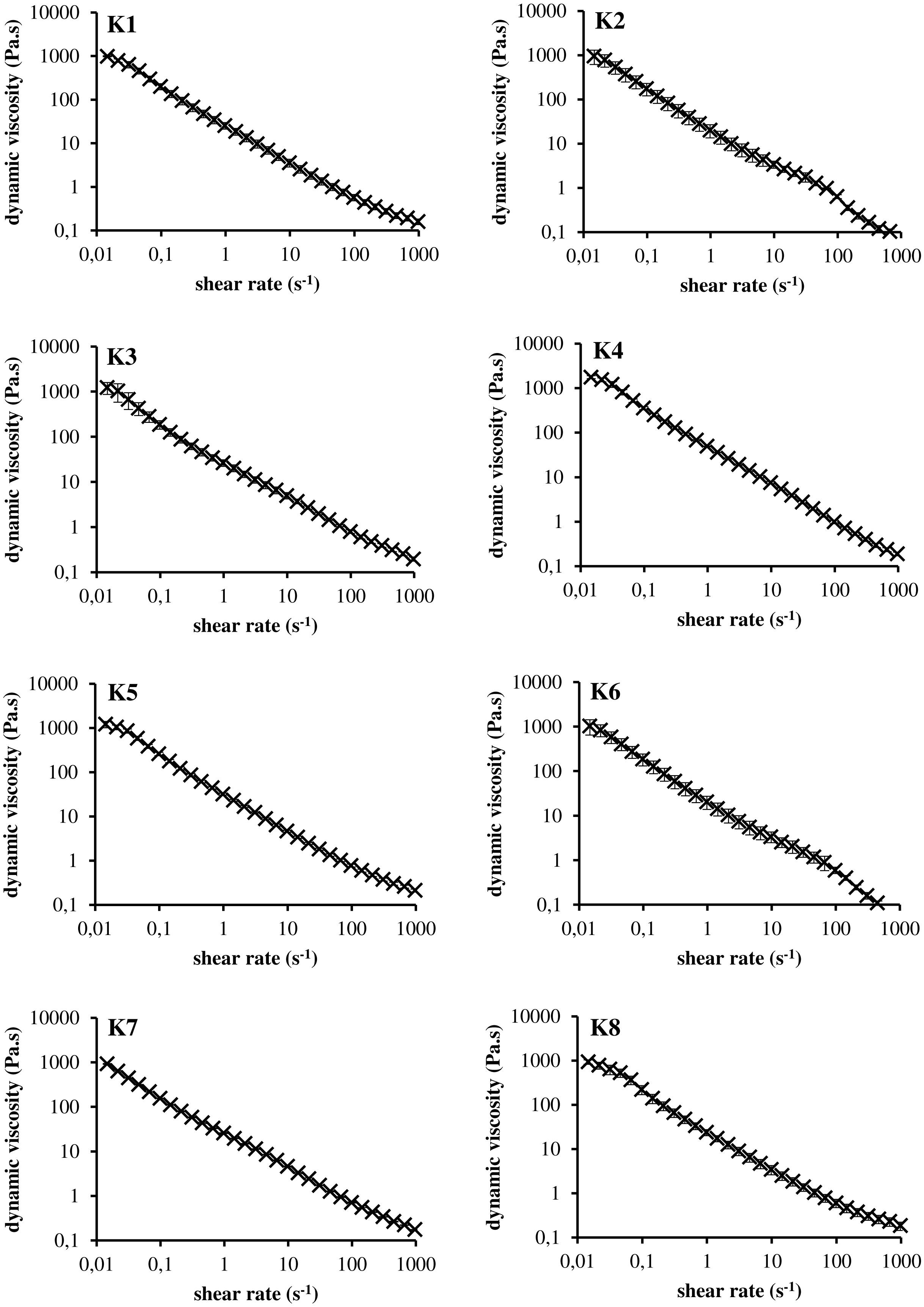

Figure 1 Flow curves of ketchup samples K1-K8.

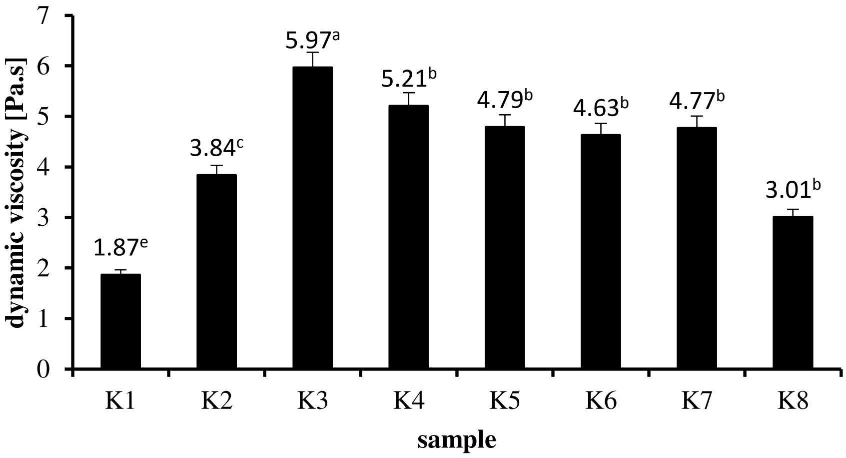

Figure 2 Dynamic viscosity of ketchup samples K1-K8. Values with different letters are significantly different at $p < 0.05$.

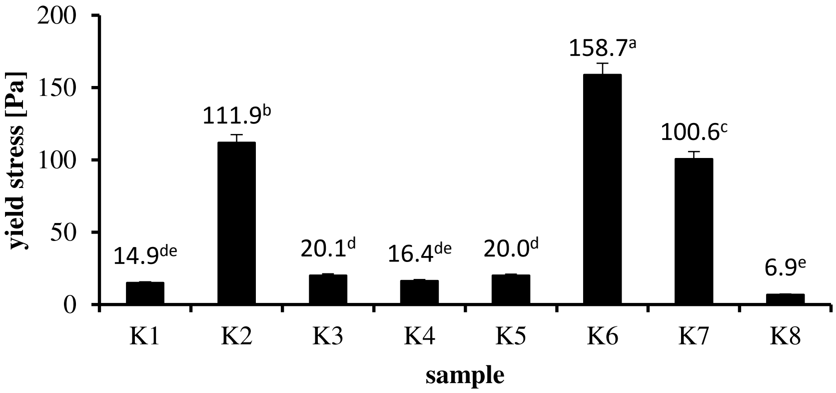

Figure 3 Yield stress of ketchup samples K1-K8. Values with different letters are significantly different at $p < 0.05$.

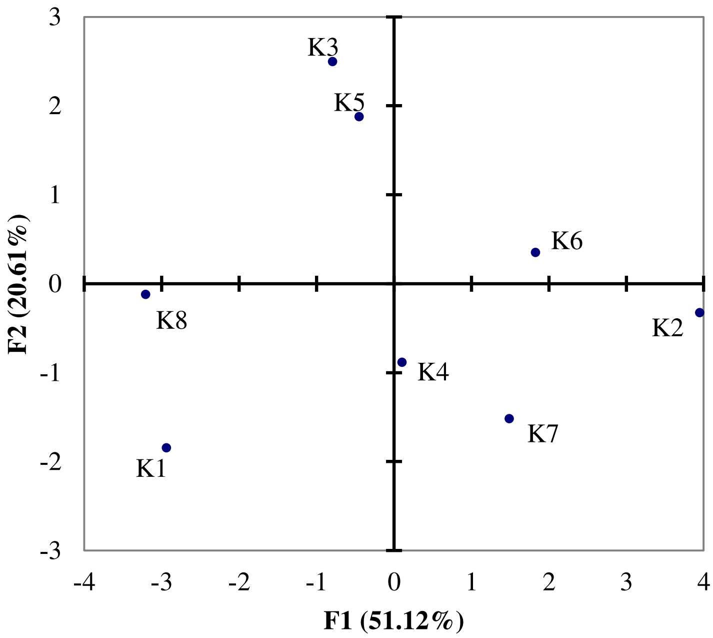

Figure 4 Plots of the first two principal components from Pearson principal component analysis of results from analysis of ketchup samples.

Tomato products are after milk, potatoes, beef, coffe, poultry, and orange juice the most important source of potassium in human nutrition **(Burton-Freeman and Reimers, 2011)**. Tomatoes into ketchup also brings a number of other nutritionally important elements which can also serve as authentic markers. Concentration of potassium in ketchup samples was within the range 1.759 – 4.979 $mg.g^{-1}$ **(Table 4)** and correlated well with tomato content in ketchup (R = 0.8864) **(Table 5)**. Concentration of other mineral elements (Ca, Mg, Na) was as follows: Ca from 0.217 to 0.910 $mg.g^{-1}$, Mg from 0.104 to 0.367 $mg.g^{-1}$ and Na from 3.570 to 8.865 $mg.g^{-1}$ **(Table 4)**. Because tomatoes naturally contain sodium in relatively low concentration **(USDA, 2015)** it can be assumed that the main source of sodium in ketchup is the salt used to flavor ketchup. Salt amount calculated from sodium content in ketchup varied from 9.1 to 22.5 $mg.g^{-1}$ which is in line with valid legislation that sets the maximum salt content in ketchup to be 30 $mg.g^{-1}$.

Viscosity is a principal parameter when any flow measurements of fluids, such as liquids or semi-solids, are made. Viscosity measurements are made in conjunction with product quality and efficiency. All samples exhibited a similar type of behavior: non-Newtonian fluid exhibiting pseudoplasticity **(Figure 1)**. Pseudoplasticity of ketchups lies in the orientation of the solid particles in the direction of flow due to the shear rate exhibited. When comparing the absolute values, there are no significant differences in the individual samples. The only difference can be seen in samples K2 and K6, which show a slight increase in viscosity between 10 – 100 s^{-1}, and there is a sign of Newtonian plateau, which can be caused by structural changes in samples K2 and K8 when subjected to deformation (shear rate). These samples also exhibit the lowest viscosity at the highest shear rates (1000 s^{-1}). Absolute dynamic viscosity values were compared at a shear rate of 10 s^{-1} **(Figure 2)**, which should correspond to typical shear rates when extruding material from the tube (1 – 100 s^{-1}). The rheological behavior of the examined ketchup samples corresponds to the results published by **Sharoba et al. 2005** or **Bayod et al. 2008**.

Ketchup is a fluid with a yield stress, it is necessary to impose some external stress at which the liquid begins to flow. High yield stress of ketchup is not so desirable, leading to dosing problems in gravity-feed systems or an excess of residue on the sides of inverted bottles. Many products are modified to keep them flowing at very low shear stress. Samples K1, K3, K4, K5 and K8 had a low yield stress, while samples K2, K6 and K7 had significantly higher yield stress (F_{crit} = 5.99, F = 65.23, p = 0.0002). For most ketchups, the determined yield stress is consistent with the results published by **Torbica et al., 2016**. Yield stress may be influenced by the amount of tomatoes used in ketchup production and by the amount of added thickeners. A significant correlation was found between tomato content in ketchup and between the yield stress value (R = 0.8436, **Table 5**).

All measured data was processed using Pearson principal component analysis. The result of this analysis is graphically shown in the **Table 5** and in the **Figure 4**. Ketchup samples were divided into four quadrants according to their similarity. The smallest distance was recorded for samples K3 and K5 located in first quadrant and for K1 and K8 samples located in third quadrant. Ketchup samples K2, K4 and K7 were all located into the fourth quadrant, but their distance was larger here. Samples K1 and K8 are ketchups with lowest tomato content (140 $g.100g^{-1}$), samples K3 and K5 are ketchups with a moderately high proportion of tomatoes (150 $g.100g^{-1}$) and samples K2, K7 and K4 are ketchupes with high proportion of tomatoes (>170 $g.100g^{-1}$). From the Pearson correlation matrix **(Table 5)** it can be seen that in addition to the already discussed correlations of some ketchup parameters, the market price of ketchups is not significantly related to their chemical composition. Weak correlation was found only between the ketchup price and the salt content in ketchup. Cheaper ketchups contained more salt.

CONCLUSION

First hypothesis that higher ketchup price means higher quality for consumers in connection with the chemical composition of the ketchup has not been confirmed as no correlation was found between the ketchup price and chemical composition of ketchup. The second hypothesis that chemical composition of ketchup affects the rheological properties of ketchups was confirmed. Ketchups with a higher content of tomatoes had significantly higher yield stress (p = 0.0002). All ketchup samples investigated in this study suit the current legislation in terms of their quality parameters. This comprehensive study, among others, showed that chemical markers as potassium or lycopene are suitable for evaluation of tomato ketchup authenticity.

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