

Physico-chemical, colour and textural properties of Croatian traditional dry sausage (Slavonian Kulen)

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Znanstveni rad

Summary

A study of physico-chemical properties, the instrumental measurement of colour and texture was carried out on ten different brands of traditional Croatian dry fermented sausage known as Slavonian Kulen. Basic physico-chemical properties showed large variability ($p < 0.05$), especially the collagen content. This can be related to different recipes used by different producers, and different stages of maturation in the samples. Parameters related to colour showed significant variability ($p < 0.05$), especially the b^* values. The higher variability of b^* could be related to the amount and botanical origin of paprika spice used in the recipe. The textural properties of examined brands were quite similar. Some differences in hardness, springiness and chewiness were significantly correlated to salt content ($p < 0.05$).

Keywords: dry fermented sausage; characterization; traditional manufacturing; Slavonian Kulen; CIE $L^* a^* b^*$; texture profile analysis (TPA)

Introduction

The traditional Croatian dry sausages are, in almost all cases, fermented sausages that undergo a more or less prolonged process of smoking and drying–ripening before consumption. The most representative Croatian dry fermented sausage is Slavonian Kulen. Slavonian Kulen is a traditional meat product from Eastern Croatia with specific sensorial properties (smell and taste), which mainly originate from being dried and smoked, and from a long period of ripening and enzymatic and lactic acid bacteria activity. It is produced according to a traditional procedure,

from minced pork, with the addition of salt, red paprika, hot paprika and garlic. The mixtures are stuffed into natural casings (a pig's appendix, lat. *intestinum caecum*).

After stuffing, the sausages undergo a smoking and drying–ripening process. The most demanding operation in the production is the ripening stage. The coherence of the processing parameters (temperature, relative humidity and air velocity) in ripening chambers are the most important, because they have the largest impact on the sensorial properties (smell, taste, colour and

texture) of the finished product. The production of traditional Slavonian Kulen mainly takes place on small farms in small amounts and it is seasonal in character. Because of that, there is a great need for the standardization of production. Sensorial properties of dry fermented Slavonian Kulen such as colour and texture have the highest influence on the consumer's perception of quality. Similar products from Spain (Chorizo de Pamplona and Salchichón) and Italy (Felino and Milano salami) have been intensively studied for their physical-chemical composition and sensorial properties (Dellaglio et al.

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1996; Perez-Alvarez et al., 1999; Gimeno et al., 2000; Bruna et al., 2003). The results of these studies showed that the use of instrumental measurements of colour and texture are the most reliable for product characterization.

There is no existing information in scientific literature on this dry sausage, which could contribute efficiently to its characterization. The purpose of this work was to examine, for the first time, physico-chemical composition and instrumental measurements of colour and texture of Slavonian Kulen, leading to the protection of geographical indications, and receiving the protected geographical indication (PGI), according to the EU Council Regulation (EC) No 510/2006 and EU Commission Regulation (EC) No 1898/2006.

Material and Methods

Material

Ten samples of traditional Slavonian Kulen were collected from different producers in Eastern Croatia (the Slavonia region). All samples had been prepared according to the traditional processing procedures without any additives such as nitrites or ascorbic added. The traditional production starts in November and lasts until June or July. It is made from the meat of pigs of at least 12 months old and over 150 kg of live weight. Only the meat from the highest quality parts of the pig, such as the thigh, the back and some shoulder (cca. 20% of the meat) is used in the production of traditional Slavonian Kulen. After choosing the highest quality meat without a high amount of connective tissue, the meat is cut into stripes 30 cm long, 10 cm wide and 3 cm thick and put on a pierced inox/stainless steel plate and placed in a freezer until the temperature of the meat reaches -2 to -5 °C. The meat is then grinded through a grinding plate with holes of 8 to 12 mm in

diameter. Optimum pH values of the minced meat at this stage should be below 5.9. The grinded meat is then mixed with salt in the amount of 2%, red paprika in the amount of 1 %, hot paprika in the amount of 0.7% and garlic in the amount of 0.2%. The minced meat is then stuffed only into a pig's appendix (lat. *intestinum caecum*) and tied with hemp rope in a special way. This operation is called "šniranje". Thereafter, the Slavonian Kulen is smoked with dry hard wood (hornbeam, beech and its sawdust) every second day for two weeks. The temperature and relative humidity at this stage should be 18 to 20 °C and 70 to 90%. After the smoking, the Slavonian Kulen is left for the ripening stage. This stage is the longest and it should last for more than 6 months in a dark room with the temperature from 14 to 17 °C and relative humidity 70 to 80%. After this stage the Slavonian Kulen can be stored in the same conditions for an unlimited amount of time.

Physico-Chemical analysis

The samples were cut into small pieces and homogenized in a household kitchen blender. The pH level was measured in a homogenate of the sample mixed with distilled water (1:10) with pH/lon 510 – Bench pH/lon/mV Meter (Eutech Instruments Pte Ltd/ Oakton Instruments, USA). Water activity (a_w) was determined using a Rotronic Hygrolab 3 (Rotronic AG, Bassersdorf, Switzerland). The FoodScan Meat Analyser was used to determine moisture, total protein, total fat and collagen according to the AOAC 2007. 04. Salt (sodium chloride (NaCl)) was determined according to the ISO method nb.1841. All measurements are conducted at room temperature (20 ± 2 °C).

Textural analysis (TPA)

Texture profile analysis (TPA) tests were performed using a TA.XT2i SMS Stable Micro Systems Texture

Analyzer (Stable Microsystems Ltd., Surrey, England) equipped with a cylindrical probe P/20. This involved cutting samples in cubes 1cm long, 1cm thick and 1cm wide, which were compressed twice to 60% of their thickness. Force-time curves were recorded at across-head speed of 5 mms⁻¹ and the recording speed was also 5 mms⁻¹. The following parameters were quantified (Bourne 1978): hardness (g), the maximum force required to compress the sample, springiness (mm), the ability of the sample to recover its original form after the deforming force was removed, cohesiveness, the extent to which the sample could be deformed prior to rupture and chewiness (g·mm) which is calculated gumminess · springiness.

Determination of colour

Colour measurements (L^* , a^* , and b^* values) were taken using a Hunter-Lab Mini ScanXE (A60-1010-615 Model Colorimeter, Hunter-Lab, Reston, VA, USA). The instrument was standardized each time with a white and black ceramic plate ($L^*0 = 93.01$, $a^*0 = -1.11$, and $b^*0 = 1.30$). The Hunter L^* , a^* , and b^* values correspond to lightness, greenness ($-a^*$) or redness ($+a^*$), and blueness ($-b^*$) or yellowness ($+b^*$), respectively. The colour measurements were performed on Slavonian Kulen at room temperature (20 ± 2 °C).

Data analysis

Three determinations for general composition and eight for texture and colour parameters were measured from each sample. Experimental data were analyzed by the analysis of variance (ANOVA) and Fisher's least significant difference (LSD), with significance defined at $p < 0.05$. Fat, collagen, pH, salt and protein and textural parameters were subjected to correlation analysis (multivariate method) to determine possible statistical relationships between them. Statistical analysis was carried

Table 1 pH, a_w and general composition**Tablica 1** pH, a_w i osnovni kemijski sastav

Brand	a_w	pH	Fat/mast (%)	Moisture/vlaga (%)	Protein/bjelančevine (%)	Collagen/kolagen (%)	Salt/sol (%)
1	0.82 ^a ± 0.10	5.06 ^a ± 0.21	23.55 ^e ± 0.57	30.77 ^a ± 0.76	35.75 ^e ± 0.82	2.66 ^d ± 0.22	6.13 ^f ± 0.19
2	0.88 ^f ± 0.16	5.58 ^f ± 0.18	27.63 ^h ± 0.43	35.62 ^e ± 0.45	26.75 ^b ± 0.51	1.91 ^c ± 0.35	4.84 ^e ± 0.2
3	0.82 ^a ± 0.12	5.21 ^b ± 0.1	14.76 ^b ± 0.65	38.49 ^g ± 0.49	36.35 ^f ± 0.22	1.11 ^b ± 0.61	6.36 ^g ± 0.17
4	0.85 ^{cd} ± 0.17	5.19 ^b ± 0.22	20.55 ^d ± 0.82	32.43 ^c ± 0.74	42.37 ^g ± 0.96	3.18 ^e ± 0.17	4.42 ^c ± 0.1
5	0.86 ^{de} ± 0.14	5.28 ^c ± 0.19	14.05 ^a ± 0.74	39.17 ^h ± 0.23	43.17 ^h ± 0.71	1.73 ^c ± 0.11	4.84 ^e ± 0.05
6	0.88 ^f ± 0.11	5.09 ^a ± 0.03	24.58 ^f ± 0.12	34.48 ^d ± 0.71	33.95 ^d ± 0.43	1.91 ^c ± 0.25	3.76 ^a ± 0.31
7	0.84 ^{bc} ± 0.09	5.41 ^d ± 0.20	15.1 ^c ± 1.72	31.43 ^b ± 0.10	53.03 ⁱ ± 0.27	2.56 ^c ± 0.31	4.07 ^b ± 0.24
8	0.87 ^{ef} ± 0.15	5.57 ^e ± 0.15	25.07 ^g ± 0.81	36.15 ^f ± 0.53	29.45 ^c ± 0.66	1.17 ^b ± 0.59	4.68 ^d ± 0.12
9	0.83 ^b ± 0.19	5.35 ^d ± 0.22	28.5 ⁱ ± 0.67	31.03 ^{ab} ± 0.41	26.21 ^a ± 0.27	0.90 ^a ± 0.27	4.93 ^e ± 0.09
10	0.89 ^{cd} ± 0.13	5.18 ^b ± 0.14	28.84 ^d ± 1.32	37.08 ^d ± 0.29	26.73 ^g ± 0.91	1.6 ^e ± 0.19	4.11 ^c ± 0.1

* Values are means ±SD of triplicate. Values in the same column with different superscripts (a-h) are significantly different ($p < 0.05$)

out with Statistica ver. 7.0 StatSoft Inc. Tulsa, OK, USA.

Results and Discussion

Ten types of Slavonian Kulen, collected from different producers in Eastern Croatia (the Slavonia region), were analyzed in this study. pH, a_w and the basic general compositions of all the brands are given in Table 1. Variability of the pH, a_w and moisture between different brands was significant ($p < 0.05$). Values for fat, protein, collagen and salt levels showed a large deviation (e.g. C.V. among brands for collagen = 39.48%). These results showed that producers use very different recipes.

CIE L*a*b* system values are shown in Table 2. The lightness (L*) values of all brands ranged from 30.7 to 42.42, the redness (a*) of all brands ranged from 15.12 to 25.34, and the yellowness (b*) from 13.57 to 26.4. Compared to other studies on similar samples (Chorizo de Pamplona) (Anserona et al., 1997; Fernández-Fernández et al., 1998; Gimeno et al., 2000; Muguerza et al., 2001; Muguerza et al., 2002), the value of lightness (L*) was lower, but the values of a* and b* were similar. The differences in L* values can be explained by the longer ripening period of Slavonian Kulen (at least 6 months), than the Chorizo de Pamplona. Com-

pared with different types of dry cured sausage, Italian Felino salami (Dellagio et al., 1996) and Spanish Salchichon (Rubio et al., 2008) colour parameters of Slavonian Kulen showed similar L* values but lower b* value, the values of a* were similar to Italian but higher than Spanish sausage. The higher b* (yellowness) values found in Slavonian Kulen are probably related to the presence of yellow carotenoids (β -carotene and cryptoxanthine) coming from paprika, a typical spice used in Slavonian Kulen production. The higher variability of b* values found among samples (C.V. = 14.22%) could be related to the different amounts and botanical origin of this spice used in the recipes of different brands. Sensorial properties can be affected by the technological process and obviously by the composition. In general, low fat levels and high water content lead to higher a* values and lower L* values. Other studies have found that the lower the protein content, the smaller the a* value (Bloukas and Paneras 1993; Carballo et al., 1995). These researchers explained this fact by the dilution of myoglobin as a consequence of the reduced protein content.

In our study no significant correlations were found between the colour parameters and protein content.

Significant correlations were also not found between colour parameters and fat, pH, collagen or salt levels, although some significant differences were found in these components among brands (Table 1 and Table 2).

Results of the TPA parameters are presented in Table 3. It is obvious that some significant differences can be observed between different brands of Slavonian Kulen. The variability of hardness between different brands was significant (C.V. between brands was 17.24%). Only the one brand of Slavonian Kulen had hardness over the 8000 g. Very low differences in springiness, cohesiveness and chewiness between ten brands were observed (C.V. = 5.93%; C.V. = 7.67% and C.V. = 9.47%). It must be mentioned that collected brands of Slavonian Kulen were not in the same stages of ripening. During the ripening process, Slavonian Kulen loses water, and other, different fermentation processes occur. This could be one of the reasons for the variability in some texture profiles between the analyzed brands.

In comparison with the results of some other authors (Gimeno et al., 2000; Gimeno et al., 2001; Bruna et al., 2001; Bruna et al., 2003; Benito et al., 2004; Revilla et al., 2005; Salgado et al., 2005; Rubio et al., 2007; Rubio

Table 2 Instrumental colour measurement**Tablica 2** Instrumentalno izmjerena boja

Brand	L*	a*	b*
1	37.03 ^{bcd} ± 1.51	23.53 ^{cd} ± 1.51	21.01 ^b ± 2.12
2	42.42 ^f ± 1.3	24.08 ^{cd} ± 0.24	25.45 ^c ± 0.62
3	39.57 ^{cdef} ± 1.34	20.29 ^b ± 0.41	19.75 ^b ± 0.35
4	36.73 ^{bc} ± 2.71	20.04 ^{bc} ± 1.21	19.88 ^b ± 1.02
5	34.45 ^b ± 1.23	20.0 ^b ± 0.12	14.68 ^a ± 0.35
6	42.21 ^{ef} ± 1.04	25.34 ^d ± 0.31	25.23 ^c ± 0.27
7	30.70 ^a ± 1.07	15.12 ^a ± 0.71	13.57 ^a ± 1.21
8	40.50 ^{def} ± 2.71	25.34 ^d ± 0.55	26.40 ^c ± 0.25
9	38.71 ^{cde} ± 2.51	19.22 ^b ± 0.87	22.88 ^{bc} ± 1.03
10	36.37 ^{bc} ± 0.9	22.04 ^b ± 1.12	19.88 ^b ± 1.41

* Values are means ±SD of eight measurements. Values in the same column with different superscripts (a-f) are significantly different (p < 0.05).

Table 3 Texture profile analysis of Slavonian Kulen (TPA)**Tablica 3** Profil teksture slavonskog kulena (TPA)

Brand	Hardness/ Tvrdća (g)	Springiness/ Elastičnost (mm)	Cohesiveness/ Kohezivnost	Chewiness/ Otpor žvakanju (g·mm)
1	8131.98 ^a ± 471.21	0.72 ^{bc} ± 0.02	0.43 ^{bc} ± 0.01	2517.66 ^a ± 305.43
2	6059.34 ^e ± 205.71	0.8 ^a ± 0.04	0.49 ^a ± 0.04	2375.26 ^{ab} ± 211.31
3	7641.44 ^c ± 331.56	0.74 ^b ± 0.06	0.42 ^{bc} ± 0.02	2374.96 ^{ab} ± 179.67
4	4803.65 ^h ± 189.73	0.69 ^{cd} ± 0.03	0.44 ^{bc} ± 0.01	1458.39 ^d ± 202.37
5	7528.88 ^d ± 213.93	0.68 ^{cd} ± 0.09	0.45 ^b ± 0.05	2303.84 ^{ab} ± 185.32
6	5951.1 ^f ± 322.86	0.66 ^d ± 0.05	0.38 ^d ± 0.01	1492.54 ^d ± 298.54
7	7769.55 ^b ± 185.66	0.65 ^d ± 0.02	0.37 ^d ± 0.02	1868.58 ^{cd} ± 148.91
8	5991.08 ^f ± 375.21	0.72 ^{bc} ± 0.03	0.46 ^{ab} ± 0.01	1948.27 ^{bc} ± 220.76
9	7827.03 ^b ± 267.92	0.69 ^{cd} ± 0.01	0.43 ^{bc} ± 0.03	2322.28 ^{ab} ± 291.12
10	5672.5 ^g ± 192.54	0.68 ^d ± 0.08	0.41 ^{cd} ± 0.02	1581.49 ^d ± 193.87

* Values are means ±SD of eight measurements. Values in the same column with different superscripts (a-h) are significantly different (p < 0.05).

Table 4 Multivariate correlations between parameters**Tablica 4** Multivarijantne korelacije među parametrima

	Hardness/ Tvrdća	Springiness/ Elastičnost	Cohesiveness/ Kohezivnost	Chewiness/ Otpor žvakanju
Fat	-0.38	0.23	0.26	-0.13
pH	-0.08	0.45	0.53	0.23
Protein	0.25	-0.47	-0.48	-0.15
Collagen	-0.29	-0.11	-0.20	-0.35
Salt	0.57 ^a	0.52 ^a	0.35	0.79 ^a

^a Significant at p < 0.05

et al., 2008), who evaluated similar fermented sausages, such as Spanish Chorizo and Salchicon, Slavonian Kulen has higher values for hardness and springiness. Values of cohesiveness and chewiness for Slavonian

Kulen were similar to these values for the Chorizo and Salchicon.

Textural properties have been related in fermented sausages to contents of fat and salt, as well as to pH

value of fermented sausages (Cofradeas et al., 2000; Fernández-Lopez et al., 2003; Gimeno et al., 2001). The results of some studies have suggested that the hardness of Frankfurter sausages decreased with an increase in fat (Ordóñez et al., 2001; Trindade et al., 2005). Additionally, Carballo et al., 1995 showed that a decrease of fat in Bologna sausages influenced the decrease in the penetration force.

In this study, differences in fat content were significant between different collected brands of Slavonian Kulen (fat contents varied between 15.10 and 28.84%). However, results of statistical analysis showed that content of fat in Slavonian Kulen brands did not influence significantly on textural properties (Table 4).

Furthermore, results presented in this study showed (Table 4) that content of salt in Slavonian Kulen influenced significantly on texture parameters. This is in correlation with the results presented by (Gimeno et al., 2000 and Gimeno et al., 2001) which showed that reduction of salt content had statistically significant influence to hardness, cohesiveness and chewiness of Pamplona chorizo. Salt reduction also caused softer texture in bologna (Seman et al., 1980) and frankfurters (Matulis et al., 1995).

In many cases pH value has been recognized as fermentation indicator, as well as like indicator of ripening stage (Hagen et al., 2000; Revilla et al., 2005; Salgado et al., 2005). In this study, variations in pH values between ten different brands were not significant (pH values varied between 5.06 and 5.58). This suggests similar stages of maturation between collected brands. If we compare pH values of Slavonian Kulen with pH values which have been observed by (Gimeno et al., 2000 and Rubio et al., 2007), it is noticeable that Slavonian Kulen has higher pH values than Chorizo and Salchicon. This could be

explained by longer ripening time of Slavonian Kulen. It is so logical that pH value of fermented sausage has been connected to texture. However, in this study, pH value was not in significant correlation with any of textural parameters. The reason for that could be in significant variation ($p < 0.05$) in pH values between the brands, which suggests different ripening stages.

Slavonian Kulen must be produced only from meat with highest quality, such as ham and back and some shoulder (cca. 15% of the meat). In this case, collagen is undesirable in Slavonian Kulen. Contents of collagen in ten brands analyzed in this study varied from 0.90 to 3.18%. As one indicator of quality, the content of collagen in Slavonian Kulen could be also connected to its textural properties. Like a part of connective tissue, collagen could change some of the textural properties. However, correlation analysis of the results determined in this study showed that collagen content did not influence significantly on textural parameters (Table 4).

Conclusions

There were large differences in the basic chemical composition of Slavonian Kulen, especially in collagen content. This shows that producers still use very different recipes in production. Colour parameters were very similar with the exception of b^* value, which can be related to the different amounts and different botanical origin of paprika spice used in the formulations. In general, TPA showed that all analyzed brands had similar textural properties. Furthermore, the content of fat, protein, collagen as well as pH value of brands, had no significant influence to any of textural parameters with some exceptions (the content of collagen on chewiness). However, the content of salt had significant influence on hardness, springiness and chewi-

ness.

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Fizikalno-kemijska svojstva, boja i tekstura slavonskog kulena – tradicionalne hrvatske trajne kobasice

Sažetak

U ovom istraživanju određivana su fizikalno-kemijska svojstva, instrumentalna boja i tekstura deset različitih uzoraka tradicionalne hrvatske trajne kobasice - slavonskog kulena. Osnovni kemijski sastav pokazao je velike razlike ($p < 0,05$) u masenom udjelu vode, proteina, masti, te osobito kolagena. Na temelju tih rezultata se može zaključiti da proizvođači koriste različite recepture u proizvodnji i da svi uzorci nisu bili u istoj fazi zrenja. Instrumentalni parametri boje L^* i a^* slavonskog kulena bili su slični dok je parametar b^* značajno varirao ($p < 0,05$) između uzoraka, što se može biti posljedica različitog masenog udjela i botaničkog podrijetla začina paprike u uzorcima. Parametri profila teksture slavonskog kulena bili su dosta ujednačeni. Vrijednosti parametra profila teksture (čvrstoće, elastičnosti i otpora žvakanju) pokazali su statistički značajnu korelaciju ($p < 0,05$) s masenim udjelima soli u uzorcima.

Cljučne riječi: trajne kobasice, karakterizacija, slavonski kulen, tradicionalna tehnologija proizvodnja, boja, profil teksture (TPA)

Physisch-chemische Eigenschaften, Farbe und Textur von Slawonischem Kulen – der traditionellen kroatischen Dauerwurst

Zusammenfassung

In dieser Untersuchung wurden physisch-chemische Eigenschaften, Instrumentalfarbe und Textur von 10 verschiedenen Mustern der traditionellen kroatischen Dauerwurst – Slawonischem Kulen, untersucht. Die chemische Grundzusammensetzung zeigte große Unterschiede ($p < 0,05$) im Massenanteil von Wasser, Proteinen, Fetten und ganz besonders von Kollagen. Auf Grund dieser Resultate kann man feststellen, dass die Hersteller verschiedene Rezepturen bei der Herstellung benutzen, sowie dass alle Musterstücke nicht in der gleiche Reifephase waren. Instrumentale Farbenparameter L^* und a^* des Slawonischen Kulens waren ähnlich, während der Parameter b^* bedeutend variierte ($p < 0,05$) unter den Mustern, was die Folge eines verschiedenen Massenanteils und der botanischen Herkunft des Paprikagewürzes in den Mustern sein könnte. Die Parameter des Texturenprofils von Slawonischem Kulen waren ziemlich angeglichen. Die Parameterwerte des Texturenprofils (Festigkeit, Elastizität und Widerstand beim Kauen) zeigten eine statistisch bedeutende Korrelation ($p < 0,05$) mit Massenanteilen von Salz in den Mustern.

Schlüsselwörter: Dauerwürste, Charakterisation, Slawonischer Kulen, traditionelle Herstellungstechnologie, Farbe, Texturenprofil (TPA)

Caratteristiche fisico-chimiche, il colore e la tessitura di kulen di Slavonia – la salsiccia croata tradizionale di lunga durata

Sommario

In questa ricerca sono state determinate le caratteristiche fisico-chimiche, il colore strumentale e la tessitura di dieci diversi campioni di salsiccia croata tradizionale – il kulen di Slavonia. La composizione di base ha rivelato grandi differenze ($p < 0,05$) in percentuale in massa di acqua, proteine, grassi, e soprattutto collagene. In base di questi risultati si può concludere che i produttori utilizzano le ricette varie in produzione, e che tutti i campioni non erano nella stessa fase di maturazione. I parametri strumentali dei colori L^* e a^* erano simili, mentre il parametro b^* variava notevolmente ($p < 0,05$) tra i campioni, la conseguenza possibile di percentuale differente e derivazione botanica di peperone in campioni. I parametri di profilo di tessitura di kulen di Slavonia erano abbastanza uguali. I valori di parametro di profilo di tessitura (durezza di elasticità e resistenza alla masticazione) hanno rivelato una correlazione statisticamente notevole ($p < 0,05$) con le percentuali in massa di sale nei campioni.

Parole chiave: salsicce di lunga durata, caratterizzazione, kulen di Slavonia, tecnologia tradizionale di produzione, colore, profilo di tessitura (TPA)

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