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C O N T E N S

AROMA AND SENSORY CHARACTERISTISC OF SLAVONIAN PLUM BRANDY

ORIGINAL SCIENTIFIC PAPER

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ABSTRACT

Quality of plum brandy is influenced by many factors, such as: climate characteristics, soil characteristics, characteristics of plum (*Prunus domestica)* varieties and technological characteristics of manufacturing process. The aim of this work was determination of the quality of Croatian "home-made" plum brandies produced by authentic production process from the Croatian region of Slavonia.
For detection of volatile flavor components and quality of plum brandy, gas-chromatography and sensory analysis were applied.
Home-made Croatian plum brandies as distillates of *Prunus domestica* contain numerous ingredients. Some of these ingredients are desirable and important for the quality of distillates, and some are undesirable. The obtained results show that all samples had a higher content of aldehydes and higher alcohols, ethanol content was in range 43.02 – 44.5 % vol.

Keywords: plum brandy, quality, flavor

INTRODUCTION

The production of plum brandies in the Croatian region of Slavonia has a long tradition. Today plum brandies are an inseparable part of local customs and gastronomy. Production is ordinarily related to small family farms, where it is carried out using specific traditional procedures and traditional copper alem-bics. Quality of alcoholic beverages is influenced by many factors. During the production process, various elements are included, such as: climate characteristics, soil characteristics, characteristics of fruit
varieties and technological characteristics of manufacturing process.

During that manufacturing process, quality of plum brandy is influenced by many various factors, such as: characterris-tics of fruit varieties, climate characteris-tics, soil characte-ristics and characteristics of technological procedure (Tuszynski and Satora, 2003; Nergiz and Yildiz, 1997; Sablayrolles et al., 1998; Singleton, 1995; Lilly et al., 2000).
However, traditional plum brandy as a distillate of *Prunus domestica* contains numerous ingredients, some of this ingredients are very desirable (Nykanen and Suomalainen, 1983; Ismail and

Williams, 1980) and important for the quality of distillates, and some are undesirable (Crowell and Gymon, 1973; Claus and Konja, 1990).

Mainly, volatile aromatic compounds are the most important for the quality of plum brandy (Velisek et al., 1982; Filajdić and Djuković, 1973; Tešević et al., 2005). These compounds can be divided into four groups:

a) primary, which derive from fruit varieties,
b) secondary, which develop during the fermentation processes,
c) tertiary substances, which develop during the distillation process,
d) quaternary substances, which develop during the maturing process.

No single group of volatile compounds is sufficient to determine the quality of traditional plum brandy. Distillation can be observed as a chemical reactor where different reactions occur which define the quality of final product. Those reactions are functions of different quality and quantity elements (Williams and Piggot, 1983; Leaute, 1990; Ho, 1996; Guan and Pieper, 1996). Today, different types of stills have been used for production of home-made plum brandies, where distillates of different qualities with complex volatile profile have been produced.

The aim of this work was to evaluate the quality of Croatian home-made plum brandies produced by authentic production process from Croatian region Slavonia.

MATERIALS AND METHODS

Fermented pulp

Six samples of *Prunus domestica* pulp were taken from the Croatian region of Slavonia, harvest of the year 2005. Samples were produced using classical technological procedure: fermentation with epiphyte yeast culture and controlled thermal regime using outer refrigeration of fermenters with running water, with the aim of keeping the average temperature in intervals of 18-20 ^{o}C. The average duration of fermentation under these conditions was 25 days.

Distillate

Samples (6) of fermented pulp were distilled in simple copper distillation devices according to the procedure of authentic distillation procedure from the Croatian region of Slavonia (modified Charente type apparatus), as shown in the scheme (Figure 1).

The samples containing approximately 45 % vol. alcohol were taken from the middle fraction, or with recommended alcohol concentration in distillates, while the first *head* and the last *tail* fraction were not used.

All selected samples were distilled according to the same distillation protocol.

Sensory analyses

A sensory analysis of samples was performed according to the method of positive scoring with factor according to the German DLG model (Koch, 1986).

This model was based on 4 sensorial experiences, which were marked with grades from 0 to 5, including 0, while the average grade is multiplied by the significance factor (Koch, 1986)

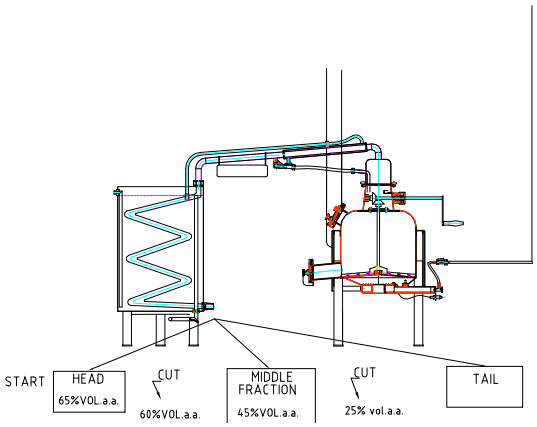

Figure 1. Scheme of equipment used for distillation of plum brandies

qualified professional testers, with extensive experience in sensory assessment of distillates, selected by selection procedure (Jellinek, 1985).

Chemical analysis of distillates

For the evaluation of the quality of distillates, fundamental analytical techniques were applied. In industrial control laboratories these techniques represent the basis for the determination of quality parameters.
Chemical analysis of the distillates included ethanol, total extract, total acidity, total SO_2, total aldehydes, total esters, higher alcohols, furfurol and methanol analysis (AOAC, 195).

Analyses of aroma substances

Gas chromatography (GC) analyses were performed on a Chrompack 437A gas chromatograph with a split/splitless injector and an FID detector. For analysis of distillates a Chrompack Poraplot capillary column (25 m x 0.25 μm i.d. 0.25 μm) was used. Initial oven temperature was kept at 35°C for 7 min then raised to 10° C/min to 80°C followed by 25°C/min to 180°C, and kept for 4 min at 180°C. Qualitative analysis was done by comparison of retention times of standards (analytical grade from Merck, Germany) and the corresponding peaks of samples. The quantification was carried out by comparison of the areas of peaks to those of the internal standard.

RESULTS AND DISCUSSION

Table 1 presents results of distillates sensory analyses. Total sensory assessment ranged in the interval between 80.7 and 98.5; which indicates significant differences (P=0.05) among samples (Dawies, 1964). Furthermore, they can be addressed to different characteristics of distillation process (differences between apparatus), since other factors in the production of distillates were the same for all samples. According to the estimated sensory characteristics, the samples 1 and 2 were evaluated as ones with the best organoleptic properties and more expressive flavor.

Table 1 Results of distillates sensory analyses of plum brandies

Sample	Color (max 15 points)	Clearness (max 15 points)	Odor (max 25 points)	Taste (max 45 points)	TOTAL (max 100 points)
1	15.00	15.00	24.50	44.00	98.50
2	14.70	14.70	23.50	43.80	96.70
3	14.70	14.40	22.50	34.00	85.60
4	14.40	14.70	21.50	30.10	80.70
5	15.00	15.00	21.50	33.00	84.50
6	14.70	15.00	22.00	34.10	85.80

Chemical analyses

As shown in Table 2, there were no larger differences among chemical and physiochemical properties of plum brandies. Obtained results were within referential values (Vila et al., 1998).

Ethanol content is very important for the mouth-feel and flavor of alcoholic beverages, lower content of ethanol (optimal 45.0 % vol.) may cause the reduction of some aroma substances in distillates (Nykanen and H. Suomalainen, 1983; Conner, 1998). Ethanol content of samples was in range 43.02 – 44.5 % vol.

The content of total extract in all distillates was within the recommended values.

The content of SO_2 in distillates ranged from 1.85 to 2.05mg/L. SO_2 was added during fermentation for the protection of pulp from non-controlled fermentation process, such as browning and oxidation. Free SO_2 may bind acetaldehyde which develops during distillation and can result in too intensive and stuffy odors of distillates (Leaute, 1990; Conner et a., 1998).

The obtained results (Table 2) show that all samples had a higher content of aldehydes and higher alcohols. Content of aldehydes and higher alcohols, identified in samples may cause formation of larger quantity of acetal during maturation of distillates. They should bring development of pleasant aroma, without sharp alcoholic odor tones.

The results of the physiochemical analysis show that the distillates have satisfactory quality, especially samples 1 and 2.

Table 2 Chemical analyses of distillates

	Sample					
	1	2	3	4	5	6
Ethanol (% vol.)	44.50	44.21	44.08	43.02	44.34	43.82
Total extract (g/ L)	0.064	0.034	0.004	0.002	0.004	0.003
Total SO_2 (mg/L)	2.05	1.85	1.89	2.04	1.98	2.03
Total acidity (mg/L)	447.60	185.00	184.20	147.00	184.20	247.00
Aldehydes (mg/L a.a.)	130.00	105.00	174.00	170.00	168.00	159.00
Esters (mg/L a.a.)	2378.23	1028.23	897.80	933.10	997.80	983.10
Higher alc. (mg/L a.a.)	1162.14	1130.71	1345.27	1537.39	1245.27	1337.39
Furfural (mg/L a.a.)	0.006	0.001	0.002	tr.	0.001	tr.
Methanol (mg/L a.a.)	0.08	0.09	0.16	0.18.	0.12	0.12.

*tr. - traces

Volatile substances (aroma substances) have dominant impact on the quality of plum brandies which were analysed with gas chromatograph (GC).

Table 3 shows the content of important aroma substances of analysed samples. According to the estimated chromatographic characteristics, larger differences among distillates were found, but all results are in accordance with the results published in previous studies (8-14). Higher concentrations of ethyl esters and higher alcohols were observed in distillates made according to the procedure of authentic distillation process from the Croatian region of Slavonia which corresponds to results published in literature (Nykanen and H. Suomalainen, 1983). Samples 3 and 4 had a higher amount of volatile substances with acetic acid ester group, such as, isoamyl acetate, 2-methylbutylacetate, and benzyl acetate, responsible for the flowery and fruity aroma (Tešević et al. 2005). Higher concentrations of aromatic terphens and higher aliphatic aldehydes responsible for plum brandy-like flavor (Crowell and Gymon, 1973) were detected in samples 5 and 6.

Table 3 Aroma compounds in distillate samples (mg/L)

Aroma Compounds (mg/L a.a.)	Sample					
	1	2	3	4	5	6
acetaldehyde	37.15	33.76	54.11	48.58	52.11	49.58
Nonalal	16.60	17.33	15.02	14.13	15.09	14.12
isobutyraldehyde	21.02	28.03	21.36	18.69	21.36	18.69
Hexanal	25.03	29.78	25.01	14.07	25.01	14.07
heptanal	19.32	18.33	16.55	15.29	16.58	15.39
1-propanol	64.75	64.70	118.20	121.00	118.20	120.00
1-butanol	0.65	n.i.	9.85	9.55	9.86	9.56
1-hexanol	5.22	2.93	2.92	1.71	2.93	1.72
isobutyl alcohol	23.62	31.39	55.37	60.42	55.38	60.43
isoamyl alcohol	148.79	139.9	88.80	115.39	88.17	115.29
2-methyl-butanol	46.32	49.00	112.01	123.02	112.00	123.00
2-phenyl ethanol	2.97	4.52	3.45	4.75	3.47	4.77
ethyl hexanoate	2.47	1.57	n.i.	2.34	1.58	2.20
ethyl acetate	16.57	14.20	44.31	57.49	44.42	57.48
isoamyl acetate	31.32	28.70	38.10	37.90	28.18	27.98
2-methylbutylacetate	18.25	19.32	20.50	20.09	16.58	17.10
benzyl acetate	21.83	22.85	23.97	23.03	20.8	21.02
ethyl lactate	3.07	1.41	n.i.	n.i.	n.i.	n.i.
ethyl octanoate	2.92	2.69	2.92	4.18	2.96	4.19
ethyl decanoate	1.23	1.51	1.68	1.74.	1.69	1.76
benzyl acetate	3.60	3.50	3.80	3.66	3.80	3.66
Linalool	0.98	0.70	0.36	0.57	0.36	0.57
α-terpineol	0.14	0.17	0.13	0.14	0.18	0.19
geraniol	0.08	0.09	0.08	0.07	0.10	0.09
Nerol	0.20	0.17	0.16	0.13	0.18	0.14
p-cymene	0.28	0.33	0.22	0.25	0.23	0.27

n.i. – not identified

CONCLUSIONS

The continental region of Republic of Croatia, Slavonia is known by production of traditional plum brandies. Research showed that plum brandies of the highest quality can be produced using specific varieties of distillation pot and specific traditional procedures. Additionally, obtained results showed, in some samples, lower concentrations of undesirable compounds such as, methanol, esters and some higher alcohol.

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THE POSSIBILITY OF APPLYING SEWAGE SLUDGE TREATMENT PLANT FOR TREATMENT OF WASTE WATER OF SREBRENIK IN PROCESS OF ANAEROBIC DIGESTION (PRODUCTION OF BIOGAS)

ORIGINAL SCIENTIFIC PAPER

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ABSTRACT

This paper examines the possibility of application of sewage sludge after the biological treatment of urban waste water in process of anaerobic digestion. Studies were conducted on sludge using water purification plant in Srebrenik city.

Characterization of physico-chemical and microbiological characteristics of sludge was performed by determination of dry matter (DM), ash content (AC), volatile organic matter (VOM), pH-value (pH), electro conductivity (EC), total amount of nitrogen (N), ammonium nitrogen (N - NH_4^+), phosphorus content (P), chemical oxygen demand (COD), heavy metals (Cd, Cu, Hg, Ni, Pb, Zn, Cr, Mo, As, Co, Al, Fe, Mn), and total number of coliform bacteria.

Concerning the amount of sludge generated on the water purification plant in Srebrenik city, as well as already analyzed sludge parameters, sludge can be subjected to the process of anaerobic digestion with the possibility of obtaining 108 247.32 m^3/god biogas, i.e., 240 MWh electricity and 343 MWh of heat.

Key words: sewage sludge, sludge, anaerobic digestion, inhibitors of the process, heavy metals

INTRODUCTION

A biological method of wastewater treatment removes over 90% of organic matter of suspended particles. In the process of wastewater treatment plant as a result we have quantities of silt which represents the excess of microorganisms and it mostly contain higher amount of organic matter. As the sludge is, due to the presence of organic matter, subject to further elaboration of the outdoors in uncontrolled conditions, there would be a separation of undesirable gases (methane, hydrogen sulfide, ammonia etc.). Also, due to the presence of pathogenic microorganisms and bacteria, which represent a potential source of infectious diseases, it is necessary to make sledge environmentally friendly and reduce the volume before the use or final disposal. A particular problem in the processing of sludge represents large water content in the separated sludge, which requires a relatively large facilities for sludge treatment (Ljubisavljević and sar., 2004).

To this end, the sludge processing is performed, which according to the schedule of processing and final disposition can be up to 30% of the total cost for wastewater treatment. Regardless of the selection process, sludge treatment aims to reduce the volume of sludge, due to cost reduction, and stabilization of the sludge, due to the prevention of natural

decomposition of sludge and destruction of parasites present in the sludge.

Unlike the industrial wastewater treatment, sludge generated from the urban waste water, should be advantageous for different types of processing and use, mainly because it does not contain heavy metals and toxic substances.

Treatment of sewage sludge using the anaerobic digestion at mesophilic conditions leads to the conversion of sludge and change its characteristics, because the anaerobic conditions degrade everyone biodegradable components, thus reducing the tendency of sludge to rot, and comes to the destruction of pathogenic microorganisms.

The conditions prevailing in anaerobic reactors are set up to encourage the development of methane bacteria. In this way, the sewage treatment plant produces a considerable amount of biogas. At the end of the anaerobic process, sludge occurs with a low content of organic matter, without the presence of harmful microorganisms, and is a matter which is suitable for soil conditioning in terms of water retention and stimulating the development of flora autotrophic microorganisms in the country, since it directly affects inorganic plant nutrients origin.

According to the above mentioned, the main objective of this work is to be based on qualitative and quantitative characteristics of sewage sludge after the biological treatment plant for wastewater treatment of Srebrenik city and to explore the possibility of its application in the process of anaerobic digestion.

MATERIALS I METHODS

Materials

As a material for analysis, sludge was used from water purification plant of Srebrenik city, which occurs in the plant as a byproduct after the biological treatment of wastewater.

Methods

Methodology for the analysis of physico-chemical and biological characteristics of sludge.

Determining the content of dry matter, ash content and volatile organic substances is performed by *Standard Methods for the Examination of Water and Wastewater*. APHA, Washington, DC (1995).

Electrometric measurement of pH was carried out according to standard ISO method 10523:1994 *Water quality - Determination of pH*. International organization for Standardization (1994), while the electro conductivity is determined by ISO method *7888:1985 Water quality - Determination of electrical conductivity,* International organization for Standardization (1985).

Ammonia nitrogen was determined by Kjeldahl (Čoha, 1990), and the value is read on the spectrophotometer VARIAN 634. Total nitrogen was determined as the sum of ammonia and organic nitrates. Nitrogen was determined by spectrophotometry with sodium salicylate, nitrite nitrogen was determined spectrophotometrically with alpha-naphthylamine - *Standard methods 4500 – N –org B*, Thermoscientific (2005).

COD was analyzed by dichromate open reflux method with the „*Standard methods for examination of water and wastewater*“ (APHA), 1995.

Phosphorus was determined spectrophotometrically by the standard *ISO method 6878:2004 Water quality - Determination of phosphorus - Ammonium molybdate spectrometric method*, International organization for Standardization (1985), and the value is read on the spectrophotometer VARIAN 634.

The content of heavy metals to the legislation of the Federation of Bosnia and Herzegovina, which treats all sludge produced from municipal wastewater (Rules for determining the allowable quantity of hazardous substances in soil and methods of their testing) is determined by atomic absorption spectrometry on the device VARIAN AA 200 (Pb, Cu, Ni, Zn, Cr, Co, Fe*, Mn* i Al*), and plasma technique on ICP – OES Perkin Elmer OPTIMA 2100 DV (Hg, Cd, As i Mo). Preparation for detection of heavy metals was done by digestion of mixture of HCl and HNO_3 on ISO 11464:1994.

The total number of coliform bacteria was determined by the method of the most probable number in 1000 ml sample.

RESULTS AND DISCUSSION

Capacity of a plant for treatment of urban wastewater in Srebrenik city is 12 000 ES and the average production of sludge is 30 m3/per day with a moisture content above 95%. Since sludge is actually waste of the rest of the plant, and concerning the fact that it contains a large amount of water it is necessary, before disposal of sludge, to concentrate and subsequently transport and dispose it at the city dump, which is a huge expense for the plant (cca 18 000 KM per year). Researched by-product (sludge) - if we want to exploit it through alternative waste through an anaerobic process of obtaining biogas must meet the basic parameters of the process and must not contain material that represents the inhibitors of the process in quantities greater than allowed.

The results of tests of physical and chemical characteristics of sewage sludge are shown in Table 1. The content of dry matter in a controlled process of anaerobic decomposition, according to Hecht-in, (2008) should be less than 20% for the wet digestion process. As the waste sludge has a dry matter proportion of 3% in Srebrenik city, before their concentration, it can be subjected to the process of anaerobic decomposition. Ash content is an indicator of inorganic compounds that are essential as microelements for the process of anaerobic digestion and ash content in sludge indicates a quite sufficient amount of inorganic matter. Volatile organic compounds are very important parameter because it is directly related with the production of biogas and methane content in biogas, as a percentage of 70.5% represents a good starting point for further research. According to Tušar (2007) the production of biogas on the basis of 1 kg of VSS is between 0.25 and 0.65 m^3 of biogas with methane content of approximately70 %. This indicates that the wastewater treatment of water in Srebrenik city was possible to get between 198 and 395 m3 of biogas on a daily basis. If we take the mean value, the production of biogas would be 296.5 m3/day or 108247.32 m3/per year.

The pH value of the material is a sensitive parameter of fluid digestion (Madamwar and Mithal, 1986). The optimal pH value for the bacteria that carry out anaerobic

*Metals which are not treated by the Regulations of the Federation

digestion is between 6.5 and 8 (Gujer and sar., 1983). As it is in our case determined by a slightly alkaline reaction of sludge (pH = 7.69), it can be concluded that the pH value is within the limits of tolerance.

Determination of electro conductivity (κ) in the sludge had a task to determine the total amount of salt in solution. The value of the electro conductivity is 1233 μS. When it comes to nutrients and the ratio COD: N: P (100:1.36:0.4), which according to the literature data (Ljubisavljević and sar. 2006) should be 100:1:0.2, in our case the ratio COD: N: P shows higher concentrations of phosphorus for a double value. However, the phosphorus content in the substrate does not present a risk of inhibiting the process of anaerobic digestion. The bigger problem arises with the concentration of ammonium nitrogen of 570.1 mgN / l, which indicates that anaerobic conditions rule in the sludge. However, the value of ammonium nitrogen will not inhibit the process, because the boundary of inhibition of ammoniac nitrogen is over 3000 mgN / l. (Chen and sar., 2008).

Furthermore, in the studied sludge the amount of heavy metals was also monitored. The values of concentrations of heavy metals covered by regulations of the Federation of Bosnia and Herzegovina, which treats sludge, and all products from urban waste water according to the rules on establishing the permissible amounts of hazardous substances in soil and methods of testing (As, Cd, Co, Cu, Hg, Mo, Ni, Pb, Zn) and three metals (Al, Fe, Mn) that does not fall into these regulations, and are significant in its characteristics to characterize the sludge, are given in Table 2, as well as maximum allowable concentrations of heavy metals according to the above mentioned Regulations.

Table 1. Physico-chemical characteristics of sewage sludge

parameter	value
dry matter content [g/l]	31.16
% dry matter	2.97
Ash content [g/l]	9.192
volatile organic substances [g/l]	21.968
% VSS into SS	70.5
pH	7.69
Electro conductivity [μS]	1233
HPK [mgO_2/l]	23400
phosphorus [mg/l]	171.6
Total amount of nitrogen [mg/l]	578.96
Ammoniacal nitrogen [mg/l]	570.1
COD : N : P	100:1.36:0.4

Table 2. Value of heavy metals in sludge and the MRL according to the

Regulations of Federation of Bosnia and Herzegovina

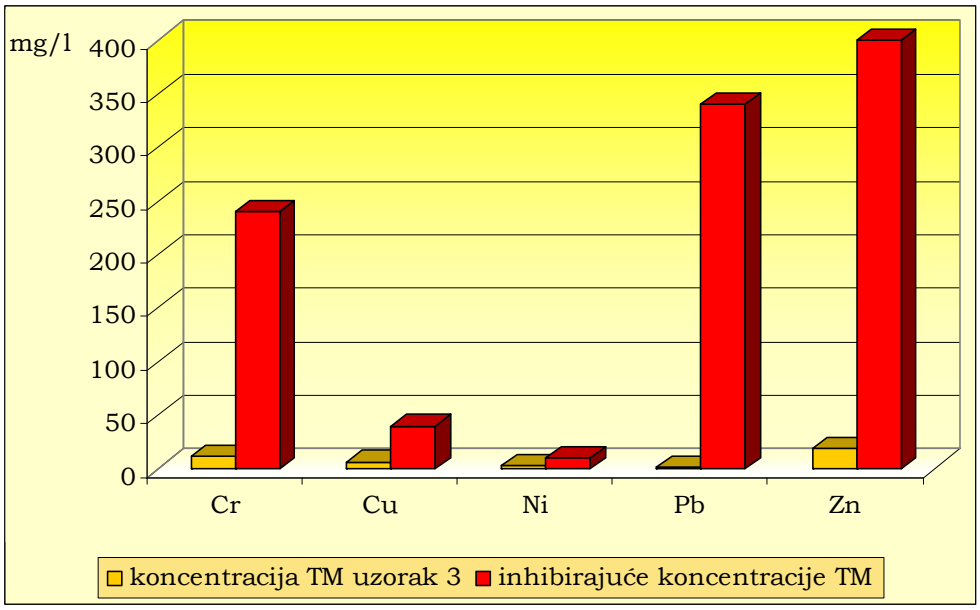
element	sample	MRL
As – arsenic [mg/kg]	19.9	20
Cd –cadmium [mg/kg]	2.139	5
Co – cobalt [mg/kg]	8.109	100
Cr – chromium [mg/kg]	368.65	500
Cu – copper [mg/kg]	202.5	500
Hg – mercury [mg/kg]	4.696	5
Mo – molybdenum [mg/kg]	<0.1	20
Ni – nickel[mg/kg]	76.8	80
Pb – lead [mg/kg]	32.3	500
Zn – zinc [mg/kg]	596.9	2000
Al – aluminium [mg/kg]	5854	-
Fe – iron [mg/kg]	1921	-
Mn – manganese [mg/kg]	900.4	-

Determination of heavy metals is important because of methane performance methane bacteria in anaerobic digestion (Voća and sar., 2004). All methane bacteria for their successful development require relatively high levels of iron, nickel, and cobalt (Ram and sar., 2000), and a sufficient amount of manganese, copper and selenium (Hecht, 2008). Optimal concentrations of iron, nickel and cobalt by different authors have completely different values (Basilko and Yavitt, 2001; Gonzales-Gill and sar., 1999; Jarvis and sar., 1997; Kida and sar., 2001; Mochinaga and sar., 1997; Schonheit and sar., 1979; Takashima and Speece, 1997). Such differences may be explained by the presence of different types of methane bacteria in the substrates that have different and unique needs for iron and cobalt. For this reason, the lack of some of these metals can lead to limitation of the entire process of production of biogas. In contrast to this, the higher concentrations of metals can cause toxicity, i.e. it prevents the development of methane bacteria (Nies, 1999). In Table 2, we can see that all the metals included in the Regulations within tolerable limits, taking care to emphasize the relatively high value of the concentration of mercury and arsenic. Due to the fact that, according to literature data, the concentrations of mercury and arsenic do not pose a risk of inhibition of

anaerobic degradation, sludge which was analyzed for digestion and biogas production at the plant in Srebrenik city, is a good basis for further research in this direction. However, concentrations of arsenic and mercury will be a problem when disposing waste residue left after any anaerobic treatment of sludge and its continued use. Special attention when talking about heavy metals should be also paid to the chromium, copper, nickel, lead and zinc, which, according to Bonmati (1996) represents a danger because of inhibiting the process of anaerobic digestion in certain concentrations, as follows:

- chromium above 240 mg/l
- copper above 40mg/l
- nickel above 10 mg/l
- lead above 340 mg/l and
- zinc above 400 mg/l.

If we compare the concentration above which there is a risk of inhibition of anaerobic processes with values obtained by analyzing the sludge from the plant in Srebrenik city compiled in [mg / l], it is not difficult to conclude that these metals will not inhibit the process (Picture 1)



Picture 1. High metals concentration relations (HM) in the sludge and the inhibiting concentration of HM

Regarding the microbiological characteristics of sludge, i.e. the number of coliform bacteria in 1000 ml sample in the tested sludge, exists more than 380 000 coliform bacteria. Coliform bacteria do not affect the process of anaerobic digestion, and that the indigenous of total coliform bacteria reduces for 97.94 - 100% in the process of anaerobic digestion for 20 days (Côté and sar., 2005), so that no waste residue behind, after the anaerobic treatment of sludge, is not burdened with coliform bacteria and does not represent a potential danger to the environment.

CONCLUSION

Based on our own research the characteristics of sludge from treatment plants of urban wastewater in Srebrenik city can be determined with:

With chemical analysis of sludge was found slightly alkaline reaction, which amounted to 7.69 as the optimal pH for the bacteria that carry out anaerobic digestion. Chemical analysis of sludge showed a slight alkaline reaction, which was 7.69, as the optimal pH value for the bacteria that carry out anaerobic digestion. The content of dry matter was low, only 3 %. 70.5 % is organic matter which resulted as a high content of organic carbon.

The high content of ammonium nitrogen in the substrate indicates the prevailing of anaerobic conditions, while the ratio COD: N: P is in accordance with literature data for the process of anaerobic digestion.

Analysis of heavy metals led to the conclusion that, in accordance with the Regulations when determining the allowable quantity of hazardous substances in soil and methods of testing ("Official Gazette of FBiH", number 72/09), none of the ten elements treated does not exceed law prescribed limits. Also, the concentration of chromium, copper, nickel, lead and zinc, which are potential inhibitors of anaerobic digestion processes at certain concentrations, will not pose a danger that the process starts in an undesirable direction.

Microbiological analysis of the sludge is determined by a high number of coliform bacteria, however, during the process of anaerobic digestion of sludge is possible to remove 97.94% ou of 100% of coliform bacteria.

The sludge, analyzed from plants for wastewater treatment of Srebrenik city, represents a waste stream and requires additional costs for processing and storage. Based on the characterization of sludge, it can be concluded that the sludge can be anaerobically digested in mesophilic conditions. Using the anaerobic digestion, it is possible to get 108 247.32 m3 of biogas a year, and from the biogas as a renewable source of energy to get 240 MW electricity and 343 MWh of thermal energy.

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MODELING OF TRACEABILITY SYSTEMS IN MEAT INDUSTRY

ORIGINAL SCIENTIFIC PAPER

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ABSTRACT

Identification and traceability in the food supply chain is a legal obligation. All participants in the food supply chain, according to current EU regulations and regulations of Bosnia and Herzegovina, which in this area are harmonized with each other, must at all times to provide a clear system of identification and two-way traceability of products. The traceability is defined as "ability to trace and follow food, feed or other substances ... through all stages of production, processing and distribution".... This applies equally to manufacturers of food, animal feed manufacturers and producers of other raw materials and ingredients, which will be incorporated into food products. Through the effective implementation of food safety and increase the efficiency of recall in case of emergency, the system of traceability provides a benefit to all stakeholders, including end consumers.

There is no clearly given instruction on how to establish a system of traceability of food. It is entirely possible to do "manualy" or on a completely automated way with the use of modern information technology. In some parts of the supply chain (for example, distribution and sale of finished products and supply of certain raw materials) for this purpose very efficiently implemented using the Global Traceability standard (GS1) system with bar code labels. However, in the production process or in the chain relating to the food industry is hardly applicable to any of the existing system. Reasons can be different: a large number of materials with different properties (size, shape, composition), a large number of transformation and mixing of raw materials in the production, the use of various technological processes, different technological regimes (temperature, humidity, etc.) and so on.

To contribute to ensuring an effective system of recording and traceability in meat processing, the authors developed a working model that will allow for easy labeling and identification of raw materials and finished products. This paper presents a model system of traceability in the case of production of cooked sausage.

Keywords: traceability, meat processing, cooked sausages

INTRODUCTION

Faced with multiple crises related to food safety, such as BSE, foot-and-mouth disease, avian influenza, the presence of toxic substances in products (for example dioxins) or genetically modified foods, companies in the food industry at the turn of the century developed new methods that will contribute to limiting the risk of new diseases, and that should help companies to convince consumers of the safety of their products (Dupuy et al., 2005).

Given the numerous crises in the supply of food, traceability has become an important issue for most food companies (Latouche, et al., 1999; Grujić et al., 2011a).

Apart from these reasons related to safety of products on the market, there are other interests to establish an effective system of traceability in the food industry (Grujić et al., 2011a, b). Shown is a marketing interest to companies through an effective system of traceability convince consumers by quality-labels of their products. Furthermore, today many companies produce products that are sold under the brand name or manufacturer named.

A good traceability system gives priority to the negotiation of it enhances the credibility of the manufacturer (the speed of response, accuracy of identification products, etc.). Even if the traceability system does not improve the quality of products, interest in the company for its implementation is bound to determine the quality of the company by tracing products, production processes and quality controls. Traceability system also helps with compliance and may affect the form of future development of the law. Finally, an effective traceability system should enable the avoidance of unnecessary and unnecessary repetitions of measures on products. If done effectively measuring the characteristics of raw materials and if you follow an efficient batch production, they are not necessary measurement properties of the products.

Moe (1998) noted the following benefits of establishing internal traceability in manufacturing companies: the ability to increase production control; indicators for determining the existence of a relationship of cause and effect in case of conflict between product, cost containment in case of mixing products with good and poor quality, easy to find information in case of audit quality and easy installation of information systems (production management, storage, quality ...). Although traceability system provides various kinds of benefits for the company, it is difficult to assess its impact on investment. In case of emergency by the food crisis establish an effective system of traceability will become the dominant benefit and reimburse all "bad investmen-ts". A good traceability system will reduce the likelihood that there will be a crisis on food security, but it can affect and reduce the consequences of the crisis. In case of crisis, the company must react quickly, accurately and reliably, they are representing the three most important characteristics of a good traceability system.

In the event of a crisis come to the fore a variety of uses well established traceability system: reduction of costs (staff and time) to explore the history of the product (determining cause and origin of the problem), cost reduction of product recall (reduces the amount of processed products to be returned to „finishing“ from the warehouse or distribution chain, the number of consumers falling ill), to companies that operate in multiple locations and/or produce more product brands, reducing the number of brands and production facilities from which to refund the product, reducing the damage due to loss of consumer confidence, the fact that the company can prove that the problem is under control.

Identification and traceability in the food supply chain is a legal obligation. All participants in the food supply chain, according to current EU regulations and regulations of Bosnia and Herzegovina, which in this area are harmonized with each other, must at all times to provide a clear system of identification and traceability of products of two-way (Regulation (EC) No 178/2002, Food Law, 2004). According to these regulations, traceability is defined as "the ability to trace and follow food, feed or other substances ... through all stages of production, processing and distribution".... This applies equally to manufacturers of food, animal feed manufacturers and producers of other raw materials and ingredients, which will be incorporated into food products. Well established traceability system makes it possible to

accurately determine changes in the composition and to determine the place where the change occurred on the product along the supply chain. Through the effective implementation of food safety and increase the efficiency of removal in case of emergency, the system of traceability provides a benefit to all stakeholders, including end consumers.

Distinguish two types of traceability. Tracing is the ability to at any point in the supply chain determine the origin and characteristics of products based on one or more specified criteria. This type of traceability is used to determine the sources of problems in product quality. Monitoring is the ability to at any point in the supply chain can find a place (localization) of the product based on one or more of criteria (GENCODE EAN France, 2001). Knowing the difference between these two types of traceability is very important. An effective information system for a kind of traceability is not necessarily effective for another.

There is clearly given instruction on how to establish a system of traceability of food. It is entirely possible to do "by hand" or a completely automated way with the use of modern information technology. In some parts of the supply chain (for example, distribution and sale of finished products and supply of certain raw materials) for this purpose very efficiently implemented using the GS1 system with bar code labels. However, in the production process or in the chain relating to the food industry is hardly applicable to any of the existing system. Reasons can be different: a large number of materials with different properties (size, shape, composition), a large number of transformation and mixing of raw materials in the making, the use of various technological processes, the processing of different technological regimes (tempera-ture, humidity, etc.).

If you create a product obtained by mixing several series of different raw materials (lots), simple traceability systems can contribute to reduce the quantity of products which, in the event of a crisis, should be revoked. The literature has been published several papers on traceability which was analyzed in product quality, traceability system developed model or a model of collecting and transferring information in the system (Dabbene and Gay, 2011; Donnelly et al., 2009; Shanahan et al., 2009). Dupuy et al. (2005), in order to optimize the system of traceability in the food industry, analyzed the batch dispersion model, or producing food products. To evaluate the accuracy of the traceability of the manufacturing process, these authors (Dupuy et al., 2002) gave a definition of three new measures: disassemble - Disintegration (downward dispersion), the preparation - integration (upward dispersion) and dispersion systems (batch dispersion). The downward dispersion of a raw material batch is the number of finished product batches which contain parts of this raw material batch. The upward dispersion of a finished product batch is the number of different raw material batches used to produce this batch. Batch dispersion of a system is equal to the sum of all raw material downward dispersion and all finished products upward dispersion. Traceability system has been successfully used during authentication (origin) of meat (Ballin, 2010).

To contribute to ensuring an effective system of recording and traceability in meat processing, the authors have developed a working model that will allow

for easy labeling and identification of raw materials and finished products. This paper presents a model system of traceability in the case of production of cooked sausage.

MATERIALS AND METHODS

The tests in this paper were performed on the example of making sausage cooked in a meat processing factory in Bosnia and Herzegovina. Preparation of samples of cooked sausage during the research was carried out according to the usual procedure of work, which applies in this company.

The system of traceability is seen as part of the supply chain, related to the fattening of pigs on farms, supply of raw materials and their reception in the company (first stage), processing / transformation of raw materials and finished goods production (second stage) and distribution of finished products on the market (third phase).

Given the existence of three phases of the traceability system in which different systems are used in labeling and identification, particularly sub-systems have been developed for "translation" tag from one system to another, or in a third system (Figure 1).

During development the most appropriate system for identification and traceability in the second stage were used three-way bill of materials for a concrete example of meat with three levels of disassembling and assembling.

Table 1. The current system of labeling and traceability in the process of cooked sausage

Documents accompanying the material at the entrance to the technological stage of production	Technological stage of production process	Documents accompanying the material at the exit of the technological stages of production
Invoice / bill of lading form suppliers VS form Microbiological analysis of meat	**Home meat (external supplier)**	Input of goods ID card with the date of receipt of raw materials The form for recording the receipt of chilled meat The form for recording the receipt of frozen meat
Work Order of slaughter Dispatch from the farm: • The number of pigs / cattle • Ear tags, list	**Home meat (slaughterhouse internal)**	Input of goods ID card materials with the date of slaughter (connection with the working order of worship)
Supplier Invoice Supplier Specification	**Admission additives, spices, packaging materials**	Input of goods ID tag with the date of receipt of raw materials
ID tag raw materials	**Storage of raw materials**	Storage Card raw materials / intermediates
Production Work Order Task recipe	**Product preparation - DISINTEGRATION, DISASSEMBLY**	Product ID card (ID code: production date) The form for recording the temperature chambers for salting and curing Preparation of chilled meat Frozen meat preparation

Documents accompanying the material at the entrance to the technological stage of production	Technological stage of production process	Documents accompanying the material at the exit of the technological stages of production
Production Work Order Working formulation Product ID card (ID code: production date)	**Mixing and filling - INTEGRATION, ASSEMBLING**	*Product ID card (ID code: production date)*
The records for the various stages of technological process Product ID card (ID code: production date)	**Heat treatment**	Product ID card (ID code: production date)
Product ID card (ID code: production date) Commercial Work Order	**Packing**	Document: products in the warehouse of finished goods Mark the package with the aggregate number of predatnice
Document: products in the warehouse of finished goods Mark the package with the aggregate number of predatnice	**Delivery of the finished goods warehouse**	Form input goods in warehouse
Customer ordering	**Receiving orders**	Commercial Work Order
Commercial Work Order	**Preparing goods for delivery**	Commercial Work Order with registered number document with packaging
Commercial Work Order with registered number of Document with packaging	**Invoicing of goods**	Customer invoices, VS form at the place
Customer invoices, VS form at the place	**Transport of goods to the customer**	Invoice signed by the customer VS form at the point of unloading Record the temperature during transport vehicles (electric)

RESULTS AND DISCUSSION

Company in which the tests were carried out particularly interested in improving the quality of its products and seeks to improve the traceability system. Pork, which is used in making various finished goods, raw materials is especially interesting in this regard. Within the company there is a modern pig farm, which is slaughtered in a slaughterhouse that is one class meat processing industry. Besides the pork that gets slaughtered pigs from our own breeding, part of the raw materials are procured from abroad or purchased in frozen / chilled state from other slaughterhouses. In addition to pork sausage cooked in the development of this company are used to a lesser extent cattle and poultry, and other ingredients (spices and food additives).

This company is a system of quality management and product safety management system a decade ago. Respecting the norms of appropriate regulations and standards, the company developed its own system of identification and traceability of products (Table 1). At first, this was a

system that required a lot of manual work of employees and filling a large number of forms which is partially slowed the performance of other activities. Later, the company opted to develop a modern system, which is supported by the information system.

The system of traceability in the meat industry consists of three phases: first phase includes the marking of live animals and monitoring of meat obtained from slaughter pigs own the farm. The second phase involves monitoring the flow of raw materials, their transformation, mixing and production of finished products. As stated in the introduction, this is the most difficult and thankless part of the implementation of traceability systems. Traceability in this section was developed as a three-level balance of material, which consists of dismantling phases and stages of preparation series / batch, which is discussed later.

The third phase of the integrated systems of traceability refers to finished products and monitoring the market. For the marking of products using the bar code system, which is acceptable in the market and what is distinctive distribution system. The first and third stage from the viewpoint of traceability of the meat industry can be seen as external traceability, and the second phase of an internal traceability.

The first phase

The first phase includes two subsystems: subsystem of marking animals from our own breeding and meat carcasses obtained slaughter of these animals and the subsystem identification and monitoring of other types of meat and other raw materials used in production. In this segment, the company follows the current domestic and EU regulations (EU, 2002; Rulebook of B&H,

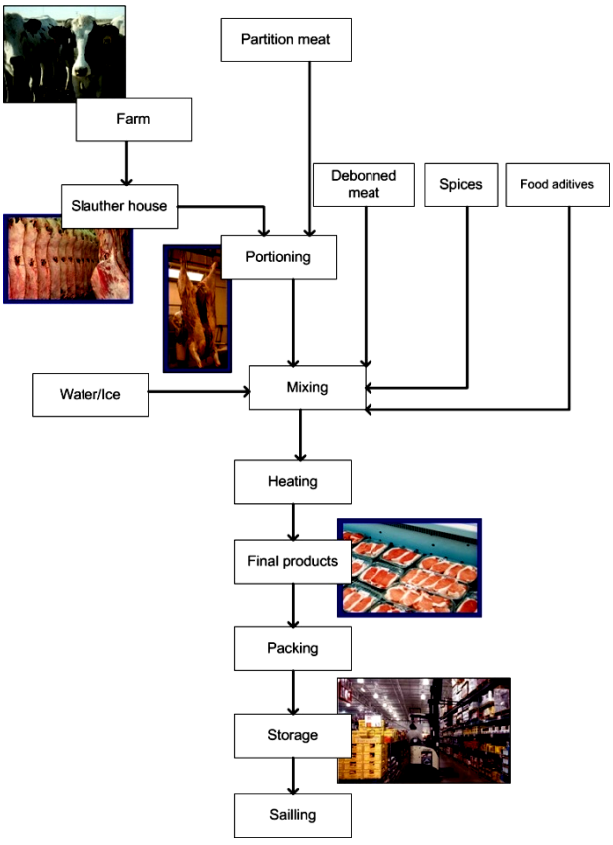

Figure 1. The system of traceability in the supply chain - such as cooked sausages

2010). Ear tag marking pigs including the designation of Bosnia and Herzegovina (BA), followed by two numbers that indicate the entity or Brčko district, followed by three letters indicating the municipality, and the next 6 numbers indicate the farm where the animal was born. Ear tag is placed on one ear, made of stainless material, not fragile and easily readable during the lifetime of the animal.

The hull is cut it into basic components (Figure 2). The company intends to increase capacity in the cutting phase of the production process: all received the series should be cut (disassemble) to the components. The first phase includes the identification and tracking subsystems chilled / frozen meat (pigs, cattle and chicken) purchased from other suppliers and identifying other raw materials used in production (spices, food additives, water).

Tagging systems resources, which are used by suppliers, differ. To avoid difficulties during the follow-up of non-local raw materials as components in the process of making finished products, the authors made at this stage of the system and uniformity of interpretation of all other systems used to own. On that occasion he used his own system of "translation" and unifying label. Raw materials that were not awarded marks are marks of their own system. The basic pieces of meat obtained from cutting their own production of hulls and pieces of meat obtained from other suppliers are marked in the same way, which is easier to track meat products regardless of origin.

The second phase

Different types and categories of meat in the production process to divide and crushed (Figure 2). Thus, the resulting pieces of meat, which is then mixed with other raw materials - components for the production of raw products and sausages. Cutting and chopping meat is the first part of the bill of material, called "disassembly / disintegration". The finished product (sausage) is obtained by mixing together multiple components in a defined relationship. This process represents the second part of the material balance of production specifications, or "assembly / integration" During the day, the company uses more sets of basic pieces of meat (ham, side of pork, shoulder ....), which were divided and mixed with other components. So, during the day in the company to use multiple sets of components of the same or different origin and are produced several series of the finished product.

The company aims to reduce the costs that may arise during the crisis caused by poor food safety. If problems related to food security comes from raw materials, the company will identify (tracing) and recall all products containing raw subject. If it is a problem of security of the finished product, the company will identify (tracking) all the products that are made from a series of raw materials and then subject them to withdraw from the market. So, in order to minimize the costs incurred during the crisis of food safety, the company should reduce the number of products that will need to withdraw from the market. In the case of sausage production that can be achieved, reducing batch size and raw materials and / or reduction of batch mixing. When mixing large batches of raw materials, then the increasing demand for the dismissal and the resulting higher costs.

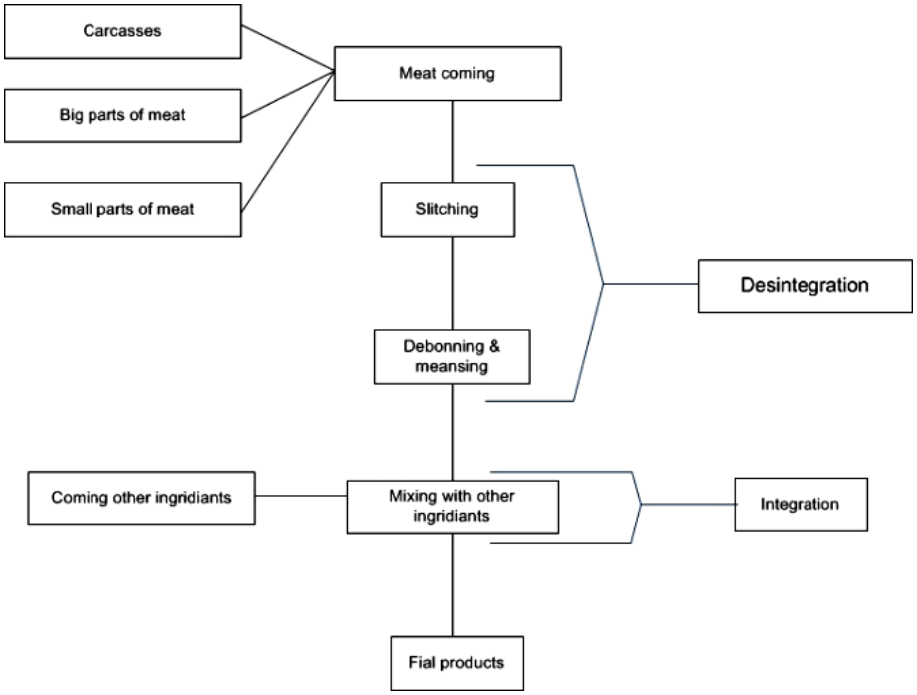

Figure 2. Three-way balance of material in the process of cooked sausage

The third phase

At the end of the process of manufacture finished products (sausages) are labeled with bar code labels, where for each batch of finished products in the internal system of traceability data is stored on raw materials, technological process parameters, quantities and prices.

Graphical model

Batch dispersion problem in the second phase of the production of sausages are not only about the process. He is present in all production processes in which there are stages of "dismantling" of raw materials and "assembly" of components in which the optimization is an important factor in traceability of product safety. The literature described more methods to solve problems created by compiling and mixing a large number of components in the development of finished products. In this regard Dupoy et al. (2005) have proposed a graphical model.

If applied in this case a graphical model to solve the problem of dispersion will be obtained Gozinto chart (Dorp, 2003; Loos, 2001) (Figure 3). Each node in the graph represents a series / batch, and each line represents the relationship between the two series / batch, if one batch / batch contains material dating from the second series / batch. The problem of dispersion series / batch to be tested, was presented at three levels: raw materials (meat), components (sliced meat, meat in pieces) and finished products (minced meat). This model allows easy visualization of the dispersions down and up.

An example from industry is characterized by a three-level balance of materials (raw materials, components and finished products).

The proposed dispersion model can be completed by adding a fourth level, which refers to the packaging process. Data Series products can be packaged in

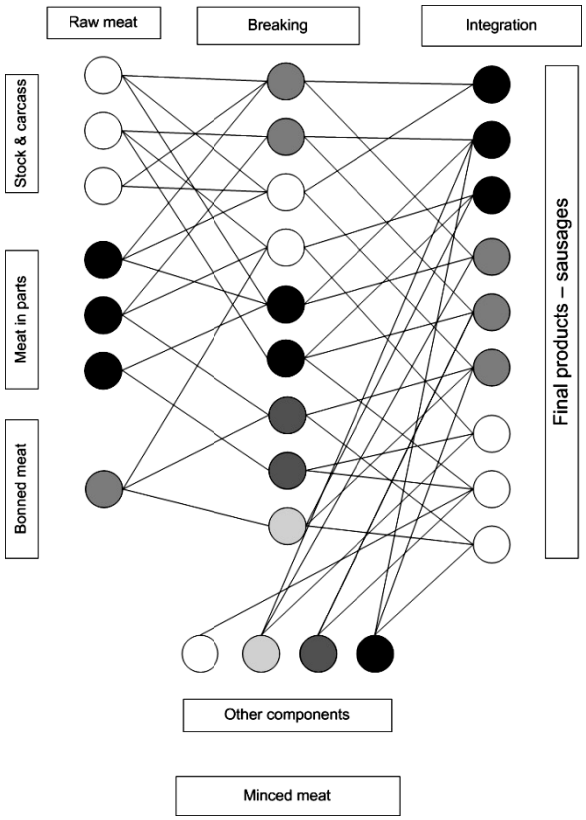

Figure 3. Graphic model of three-way balance

different ways. Since the packaged products can be drawn from different production series as, for example, sausage meat or with different spices in the same package. In that case you need to develop a mathematical model with four degrees of dispersion.

CONCLUSION

This paper presents a model example of traceability in the food industry, which is based on *three-way balance* material (raw materials, components and finished products). In case of emergency for the safety of products, it is important to reduce the amount of product that needs to be revoked. The proposed model of traceability makes it easier to identify series / batch of finished products that are built suspicious materials and thus reduces the size of the series that needs to be revoked.

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QUALITY OF HAY AT FARMS IN BOSNIA AND HERZEGOVINA

PROFESSIONAL PAPER

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ABSTRACT

The purpose of this research was to determine the quality of hay used on dairy farms, and, based on the results and analyzes of hey quality provide recommendations for improvements of animal feed in order to increase milk production per cow.
Quality of hey depends on the number of factors: type and sort of grass and / or legumes, land fertility, climatic conditions, season, stem-to-leaf ratio in grass, stem thickness, weeds, weather conditions at the time of grass cutting, technology of preparation and storage, etc. Yet, the most significant factor is the grass / legume maturity at the time of cutting.
The following quality parameters of silage hey were analyzed: dry matter, protein, fiber, and minerals. Analyzed samples had, on average, desirable dry matter content (86.59 grams in 100 grams of fresh sample), low protein content (96.43 grams in 100 grams of dry matter), high fiber (cellulose) content (44.05 grams in 100 grams of dry matter) and low mineral content (2.11 grams in 100 grams of dry matter).
Statistically significant differences were observed in dry matter content (varying from 74.15 to 91.74 grams in 100 grams of dry matter), protein content (varying from 5:52 to 3.17 grams in 100 grams of dry matter), fiber content (25.24 to 66.07 grams to 100 grams of dry matter) and mineral content varying from 0.93 to 3.87 grams in 100 grams of dry matter.

Key Words: hey, protein, cellulose, dry matter, mineral matter

INTRODUCTION

Traditional manner of conservation of green fodder is to dry it and store it in the form of hey. The purpose of preparation of hey is:

- To cut grass in the optimal development stage which ensures maximal quantity of feed without negatively affecting the other crops to be planted on the same land plots, and
- To reduce the moisture content from 65-85% to 10-15% by drying to prohibit the growth of plant and microbial enzymes which spoil the feed quality.

Hey production is predominant in preparation of bulky feed. The production of alfalfa feed is prevented by the acidic soil, which is characteristic soil type of the region. The potential replacement crop for alfalfa is red clover which tolerates acidic soil.
Another problem in production of animal feed is the technique used in natural and artificial grazing fields. Majority of farmers plant the seeds in the spring and

abstain from use of mineral or natural fertilizers. These factors contribute to low quantity of plant material and animal feed produced.

Introduction of new fodder cultures, such as fodder peas, beans and other legumes is at very poor level. A dominant grass is the perennial ryegrass (*Lolium perenne*).

Production of clover-grass mixtures occurs only in very limited quantities, on small areas. The main reason for such limited production is the lack of knowledge in appropriate production technology. Most farmers stressed that the different maturation cycles of grass and clover creates a dilemma about what is the appropriate time to harvest. Yet, as a rule of thumb, a great majority of farmers harvest the plants in their late-maturity stages due to a common belief that a quantity, rather than the quality of forage, is the more important factor in production. The forage plants are typically harvested two to three times per year. This is a very low number, if we compare the local practice with some other neighboring countries. Several red clover and clover-grass mixtures production trials have been ongoing, with an aim of improving forage production.

The goal of hay production should be to attain the highest possible quantity of milk by provision of quality forage on a given land plot, rather than to produce as large quantity of hey as possible.

Hay is characterized by low concentrations of digestible nutrients and a large amount of ballast- an indigestible organic matter (Kalivoda, 1990).

Quality of hay can be ascertained by visual inspection, taking into consideration the maturity of harvested grass, its color, smell and cleanliness. The following chemical parameters are associated with the consumption and quality of hay: energy value (Ball et al., 2002), and the content of fiber, protein and dry matter (Cherney and Martens, 1998).

Quality of hey depends on the number of factors: type and sort of grass and / or legumes, land fertility, climatic conditions, season, stem-to-leaf ratio in grass, stem thickness, weeds, weather conditions at the time of grass cutting, technology of preparation and storage, etc. Yet, the most significant factor is the grass / legume maturity at the time of harvest. By postponing the harvest from vegetative to reproductive (seed development) stage increases plant fiber (cellulose) content while simultaneously decreasing the content of protein in a plant, thus affecting the digestibility and feed intake.

The practice of mowing the grass mass at a later stage of maturity (full flowering) to facilitate preservation by drying (contains more dry matter at harvest time), and achieving a slightly higher yield of dry matter per unit area, rather than mowing a lawn in the early stage of maturity (stage of full development of leaf mass, the start of flowering), which ensures quality (Vranić et al., 2004).

According to Hoveland et al. (1997), a four-week postponement of harvest reduces protein content in hey by 4-6% and digestibility of organic matter in dry matter by 13%. Reduction of forage quality resulting by harvesting delays is associated with an increased content of lignin and structural cell wall components, i.e. decreased protein content and decreased concentration of digestible plant cell components such as starch (Aman and Lindgren, 1983).

In vegetative stage of plant development, the leaf constitutes the larger part of the plant than the stem. As the plant matures,

the leaf gets smaller while the stem grows in relative proportion to leaf's receding rate. This causes a decrease in the raw protein content and an increase in the raw fiber content (Di Marco et al., 2002).

An average cow can easily consume the following quantities of hey (assuming the cow has been fed with hey only):

- About 12 kg of low quality hey. This is a quantity sufficient to produce 2.2 liters of milk (taking into account animal's maintenance needs);
- About 15 kg of good quality. This is a quantity sufficient to produce 15.3 liters of milk (taking into account animal's maintenance needs).

Put differently, by using low quality hey in animal feed reduces potential income by a factor of seven as compared to potential income when good quality hay is used (Oresnik, 2002).

Higher fiber content reduces forage digestibility, its energy content and potential consumption, which directly affects milk production. The digestibility of fodder and "at will" consumption are being reduced in concert with decrease in forage quality. Hence, to meet the nutritional needs of highly productive animals, it is necessary to enhance their feeding regimen with other types of supplemental forage.

MATERIALS AND METHODS

The survey included hay samples from 41 farms (located in Una-Sana Canton, Banja Luka, Kozarska Dubica, Prnjavor, Bijeljina, Bratunac, Modrica, Derventa, Prijedor, Tuzla Canton, and Posavina Canton). The surveyed farms have more than 10 dairy cows. Samples were analyzed by the Agricultural Institute of Una-Sana Canton.

The chemical quality of the hay samples was determined by the following methods:

- Protein (nitrogen) - (sample preparation, digestion, distillation, titration), apparatus by Kjeldhal procedure;
- Cellulose- manufacturer's method (VELP) – cellulose extractor;
- Humidity (dry matter) - automatic moisture device (Ohaus);
- Minerals- method of burning and annealing (burner and furnace annealing).

Based on the results of analysis, consultants from the Republika Srpska Extension Services Agency, Agricultural Institute of Una-Sana Canton and Agricultural Institute of Tuzla Canton, developed recommendations on the hey feeding regimen of dairy cows.

Data were analyzed using SPSS 12 statistical program (Descriptive statistics and One Sample test).

RESULTS

Forage plants should be harvested up to the period of their full bloom, or alternatively, by the end of the blooming cycle at the latest, as the plants accumulate nutrients in stem and leaves to provide them in the process of seed development. Hence, the largest concentration of nutrients can be found in a plant immediately before the bloom. Once the flowering occurs, nutrients are being transferred to plant reproductive organs – fruit and seeds – with certain quantities of nutrients being transferred to the plant root system. As a consequence, stem and leaves become

nutrient impoverished, especially of protein, and the cellulose content increases. Hay made of plant material at this stage cannot be considered quality hey, due to resulting low nutritional quality.

Table 2 The average chemical composition of hay samples (n = 41), n –number of samples

Parameter	Mean	St. dev.	Max.	Min.
Dry matter	86.5924	3.09739	91.74	74.15
Protein	9.6429	3.28439	17.33	5.52
Cellulose	44.0522	10.64211	66.07	25.24
Mineral matter	2.1124	0.61142	3.87	0.93

Well-preserved hay should contain 85-90 grams of dry matter per 100 grams of fresh sample, which prevents propagation of microbial organisms which spoil hey. If the stored hey is wet (more than 15% moisture), the temperature increases significantly, thus reducing the quality of hay. Analyzed samples had, on average, adequate dry matter content concentration of 86.5942 grams in 100 grams of fresh sample.

On average, low protein content is an indicator of late harvest of grasses and legumes. Optimal protein content for grasses and legumes harvested in budding or flowering stages range from 10.4 to 18.4. The average protein content observed in the hay samples was 9.643 grams in 100 grams of dry matter.

The high cellulose content is correlated with protein content. Late harvest increases cellulose concentration and reduces the protein concentration. Optimal cellulose content in late-harvested plant material should not exceed 42.3 grams. Yet, the average cellulose content in the collected samples was 44.0522 grams in 100 grams of dry matter.

The mineral matter content is an indicator of soil fertility and proper fertilization of grasses and legumes. The low mineral matter content in the analyzed samples of hay (on average 2.11 grams in 100 grams of dry matter), is an indicator of poor soil fertility and inadequate application of mineral fertilizers in fertilization of grasses and legumes.

In order to ensure the quality of hay, it is necessary to do the following:

- Conduct a chemical analysis of soil every 4-5 years.
- Conduct ameliorative fertilization and liming every 4-5 years, in accordance with the observations gained from soil analysis. Soil should contain at least 16-18 mg of phosphorus and potassium with sufficient quantities of calcium.
- Prepare the land for planting and conduct fertilization for ongoing production in addition to pre-planting fertilization; 2/3 NPK fertilizer should be applied in such manner to ensure sufficient fertilizer quantity at the bottom of the furrow, and 1/3 to be applied on the top.
- Supplemental mineral fertilizers NPK 10:20:30 should be applied; $2/3^{rd}$ in the fall, $1/3^{rd}$ in late February and early March; KAN should be applied before vegetation and after each harvest.
- Procure grass seeds or alfalfa seeds.
- Harvest and storage should take place in earlier stages of maturation.

Table 3. Chemical composition of hay samples

Number	Moisture %	Protein	Cellulose	Min. matter	Dry matter
1	9,6	9,8	45,04	1,46	90,4
2	15,25	6,77	49,4	1,9	84,75
3	12,11	5,8	59,71	0,93	87,89
4	11,61	7,86	46,99	2,31	88,39
5	11,66	9,59	40,79	1,93	88,34
6	11,35	12,47	34,37	1,06	88,65
7	11,57	5,52	43,76	1,45	88,43
8	15,36	12,95	33,4	1,56	84,64
9	13,21	14,52	25,24	2,53	86,79
10	11,92	10,09	39,68	1,3	88,08
11	25,85	12,98	31,59	2,47	74,15
12	16,72	8,95	46,09	3,56	83,28
13	11,42	8,7	44,64	2,76	88,58
14	12,78	7,43	59,19	2,06	87,22
15	17,57	7,97	46,84	2,58	82,43
16	15,59	14,11	37,6	1,73	84,41
17	14,52	8,7	40,48	2,59	85,48
18	11,96	8,34	42,56	3,03	88,04
19	14,92	7,81	57,47	2,91	85,08
20	19,18	13,33	33,63	1,98	80,82
21	11,95	6,53	47,38	2,56	88,05
22	15,3	7,79	49,44	1,59	84,7
23	11,88	7,37	62,92	3,03	88,12
24	14,09	7,02	47,27	1,98	85,91
25	10,54	6,22	66,07	2,05	89,46
26	10,75	15,83	25,4	2,05	89,25
27	11,22	8,84	65,65	1,91	88,78
28	10,82	9,62	43,52	1,91	89,18
29	12,54	6,65	41,06	2,32	87,46
30	13,69	6,65	40,08	1,95	86,31
31	11,95	13,99	38,54	1,85	88,05
32	15,25	11,54	35,79	1,67	84,75
33	14,59	9,62	46,82	2,54	85,31
34	8,26	5,77	64,96	3,87	91,74
35	11,56	7,35	45,02	2,15	88,44
36	17,89	16,3	29,17	1,75	82,11
37	15,02	17,33	28,53	1,85	84,98
38	11,72	14,2	38,75	1,6	88,28
39	14,6	10,68	36,48	2,02	85,4
40	11,44	6,24	40,42	1,82	88,56
41	10,4	6,13	53,4	2,04	89,6

Table 4. Descriptive statistics and test

	n	Min	Max	Mean	St. dev.	Standard Error	T	df	95% confidence interval	
									Lower	Upper
Protein	41	5.52	17.33	9.6429	3.28439	0.51294	18.799	40	8.6062	10.6796
Cellulose	41	25.24	66.07	44.0522	10.64211	1.66202	26.505	40	40.6931	47.4113
Mineral matter	41	0.93	3.87	2.1124	0.61142	0.09549	22.123	40	1.9195	2.3054
Dry matter	41	74.15	91.74	86.5924	3.09739	0.48373	179.009	40	85.6148	87.5701

Out of the total 41 samples of hey analyzed, 11 samples had less than 85 grams of dry matter in 100 grams of fresh sample. This means that 27% of samples analyzed had high moisture content. This is an indicator of inadequate drying process. Such hey is more susceptible to deterioration in quality and consequently, reduction in milk quality produced.

Total of 13 samples, or 31.71%, had protein content higher than 10.4 grams in 100 grams of dry matter. Additionally, 21 samples, or 51.22%, had cellulose content greater than 42.3 grams in 100 grams of dry matter.

The cellulose and protein content observed in samples is congruent to Table 1, i.e. with increase in cellulose content, the protein content will decrease.

CONCLUSION

Analyzed samples of hay obtained from 41 farms were low to medium quality (low protein content, high cellulose content, and low content of mineral matter). Plant material was harvested in the late stages of plant development, which significantly differs from recommended practices for production of high quality hey for dairy cow feed.

Late harvest of plant material is indicated by the low average protein content (9.64 grams in 100 grams of dry matter) and high average cellulose content (44.05 grams in 100 grams of dry matter). The relationship between the protein and cellulose content in samples (high in cellulose, low in protein) is an indicator of plants harvested in late stages of development.

It is recommended that the plant material used in preparation of hey is harvested 4-6 times, and to produce no more than 10% of hey for animal feed from harvested quantities. The undeveloped grass land should be used for storing hey or grass silage in silos.

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CHEMICAL QUALITY AND SHELF LIFE OF COLD-PRESSED SUNFLOWER OIL PRODUCED FROM THE SEED STORED FOR 6 MONTHS

ORIGINAL SCIENTIFIC PAPER

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ABSTRACT

The objective of this study was to investigate chemical quality and shelf life of edible sunflower oil prepared by the procedure of cold pressing on a screw press from seed stored for 6 months. The seed for pressing was prepared with different contents of impurities and hulls. It was found that for production of cold-pressed sunflower oil of good chemical quality, it is possible to use seed stored under controlled and adequate conditions for 6 months, which is supported by *AV* which range from 0.23±0.01 to 1.50±0.02 mgKOH/g. Furthermore, it was concluded that also the presence of impurities (up to 10%) and hull (up to 32%) had an adverse effect on the chemical characteristics of the analyzed oil. The results of these analyses also point out to the fact that sustainability of the oil made from the stored seed was good, and that the most important effect on reduction of induction period was shown by the presence of impurities in the pressing material. The shortest *IP* of oil of 9.35±0.02 h was identified for the seed with 10% of impurities, while the sample made from pressing of seed with 32% of hull had the longest induction period of 10.88±0.03 h.

Keywords: seed storage, impurities, hull, chemical quality, shelf life, cold-pressed sunflower oil

INTRODUCTION

The quality of cold-pressed sunflower oil depends primarily on the quality of the raw material for pressing. The production of especially high quality oils is considered to be a real challenge to the manufacturers[1,2,3]. One of the main problems in the production of cold-pressed sunflower oil is the variable quality of the starting raw material[4]. The difficulty is that seed are not processed directly after harvest, but the processing takes place continuously over at least 1 year[2,3]. Therefore, seed have to be stored under optimal conditions, but seed is a living organism, with metabolic processes which result in degradation of storage compounds of seed like lipids, carbohydrates or proteins and the development of aroma-active components. Depending on the volume of metabolic processes, which are influenced by temperature, moisture and time of seed storage this can be associated with a dramatic loss of mass as a result of the degradation of carbohydrates into water, carbon dioxide and energy.

Therefore, the time between harvesting and seed pressing needs to be reduced to minimum, and all measures need to be taken in order to prevent, postpone or reduce to minimum the beginning of biochemical or chemical reactions (e.g. hydrolysis or oxidation), which reduce the quality of oil in raw material itself. In order to postpone the beginning of these

negative chemical processes, raw material must be prepared with care under adequate and controlled moisture and temperature conditions[1]. Only a short time of storage of sunflower seed under inadequate conditions is sufficient to destroy very good raw material with lasting effect[3].

While evaluating the quality of edible oils, it is important to know chemical quality and sustainability, that is their oxidative stability. Oxidative stability is especially important for oils used for frying and roasting. Due to high temperatures, long process of frying and roasting, and extremely complex chemism of oxidation at high temperatures, oils intended for these purposes must have high oxidative stability during longer periods of use[5]. It is necessary to determine oxidative stability during oil quality evaluation due to its effects on other quality parameters. The process of oxidation of lipids cause changes in chemical structure of oil, and consequently on its nutritive and sensory quality[6].

Apart from the quality and seed age, the quality of cold-pressed sunflower oils, that are widely present on the market, is especially influenced by the presence of impurities and hulls in the pressed material[3]. Bearing in mind the fact that chemical characteristics are related to the oil stability[7,8], the knowledge of the corresponding parameters is of crucial significance from the point of view of both producer and consumer of oil. In view of this, the objective of the present study was to investigate the chemical quality and shelf life of cold-pressed sunflower oil produced from seed stored for 6 months with the presence of different amounts of impurities and hulls in the starting material for pressing.

MATERIALS AND METHODS

Materials

The investigation was carried out on five samples of cold-pressed oil prepared by pressing domestic linoleic type sunflower hybrid CEPKO. The two dominant fatty acids in the oil composition were oleic 32.9 and linoleic 55.61 wt% of total fatty acids. The sunflower seed were product of conventional cultivation, cleaned and stored in silo cell under the conditions of low-temperature and good ventilation for 6 months until processing.

The experiments were carried out in a small scale oil factory equipped with seed cleaner, dehuller and screw press for processing of cold-pressed sunflower oil. Dehulling of the seed is carried out by an impact dehuller with removal of the hulls by airflow and gravity.

The oils were produced by pressing material with designed content (dehulled kernel and dehulled kernel with the addition of certain amounts of impurities and hulls) according to the following experimental design: Sample 1: dehulled kernels with 0% impurities and 0% hulls, Sample 2: dehulled kernels with 0% impurities and 32% hulls, Sample 3: dehulled kernels with 5% impurities and 16% hulls, Sample 4: dehulled kernels with 10% impurities and 0% hulls, and Sample 5: dehulled kernels with 10% impurities and 32% hulls.

Each of the samples of the above composition was pressed in duplicate using each time 5 kg of the material. The pressing was performed on a screw press (Anton Fries, Germany), capacity of 6-9 kgh^{-1}, at the rotation speed of 35-40 rpm. The temperature of the oils at the press

outlet was 55-60 ºC. The pressed oils were kept at room temperature (20-25 ºC) for 24 hours for sedimentation of residues, then the upper layer was decanted and filtered through ordinary laboratory filter paper. To their analysis, the oil samples were stored in glass bottles at 4 ºC.

Impurities consisted of usual admixtures, mainly of organic origin and fatty dust present in the seed mass. The basic characteristics of impurities were: moisture - 10.12±0.08%, acid value - 95.20±1.43 mgKOH/g and peroxide value - 23.82±0.07 mmol/kg.

Methods: Moisture content (%) was determined by conventional method by sample heating at 103±2^{o}C to the full elimination of water and volatile matter, i.e. to the constant mass of the dry residue (ISO 660:2000). The ***acid value*** (*AV*) (ISO 660:2000), expressed in mgKOH/g, was determined by titration of a solution of oil dissolved in 1:1 ethanol:ether with ethanolic solution of potassium hydroxide. The ***peroxide value*** (*PV*) (ISO 3960:2001) expressed in mmol/kg, was determined by the reaction of oil and 3:2 chloroform:acetic acid with potassium iodide in darkness. The free iodine was then titrated with a standard thiosulfate solution. The ***p-anisidine value*** (*p-AnV*) was determined following the AOCS official method (Cg 18-90), on a UV/VIS spectrophotometer, model T80+, PG Instruments Limited, London. The ***Totox value*** was calculated as twice *PV* plus *p-AnV*.

Determination of oxidative stability. The oxidative stability of oils was investigated by determining the induction period (*IP*) on a Rancimat 743 apparatus, at 100^{o}C and an air flow of 18 L/h (ISO 6886:1996). Portions of oil (2.5 g) were carefully weighed into each of 12 reaction vessels and analyzed simultaneously.

The influence of heat on the stability of oil samples was investigated using ***Schaal test***[9]. The amount of 50 g of each oil sample was placed in two open *Petri* dishes, stored in an oven at 63±2^{o}C in dark, and tested for deterioration after 96 h, where by the *PV* was determined. Stability was expressed in % as the change in peroxide value (*CPV*) according to the formula[10]: $CPV(\%) = 100\ (PV_2 - PV_1)/PV_1$; PV_1 – peroxide value of sample at the beginning; PV_2 – peroxide value at the end. All reagents used were of analytical grade (Merck, Germany).

Statistics. The experimental values were expressed as the means of four determinations (two oil samples in two replicates). Statistical analysis was performed using the Statistica 8 software package. Statistical differences between the oil samples were estimated by applying two-way ANOVA and using the Tukey test at a significance level of 5% ($p<0.05$).

RESULTS AND DISCUSSION

The effect of the 6-month sunflower seed storage on the chemical quality of pressed oil can be seen in Table 1.

The moisture contents of the investigated oil samples (Table 1) were in the range from 0.05±0.00 to 0.06±0.01% and these values are far below the maximum tolerable value prescribed by the legislation[11].

Acid value is an important parameter of oil quality as it indicates the possible damages of sunflower seed during storage. The *AV*

of investigated oils (Table 1) is low, ranging from 0.23±0.01 to 1.50±0.02 mgKOH/g, which is lower compared to the literature reports quoting the *AV* for edible unrefined sunflower oil from seed stored for 8 months, which is 3 mgKOH/g[12].

It is well known that *AV* value of pressed sunflower oil increases with the time of seed storage[13], however low *AV* values of analyzed oils show that prior to pressing sunflower seed was stored under adequate and controlled low temperatures and good ventilation[3].

The analysis of *AV* results shows that there exists a statistically significant difference ($p<0.05$) between all oil samples. Presence of both impurities and seed hulls in the seed stored for 6 months exerts an adverse effect as these components cause an increase in the oil acidity. Negative effect of seed hulls on acidity of pressed oils has been reported in the literature by Zheng et al. (2003)[14]. The lowest *AV* was measured for the oil prepared from dehulled sunflower kernel (0.23±0.01 mgKOH/g - Sample 1), and the highest for the oil sample obtained from the material with 10% impurity (1.50±0.02 mgKOH/g – Sample 4). A significant effect of higher contents of impurities in the pressed material on oil acidity was expectable since the *AV* of impurities was extremely high, 95.20±1.43 mgKOH/g.

Table 1. Basic chemical quality parameters of cold-pressed sunflower oils

Sample	Moisture (%)	AV (mgKOH/g)	PV (mmol/kg)	p-AnV	Totox
1	0.05±0.00 ^{ab}	0.23±0.01 ^{a}	1.57±0.01 ^{a}	0.00±0.00 ^{a}	3.13±0.02 ^{a}
2	0.06±0.00 ^{ab}	0.36±0.01 ^{b}	1.47±0.06 ^{b}	0.17±0.00 ^{b}	3.11±0.01 ^{a}
3	0.05±0.01 ^{a}	0.59±0.03 ^{c}	1.95±0.01 ^{c}	0.00±0.00 ^{a}	3.89±0.03 ^{b}
4	0.06±0.00 ^{ab}	1.50±0.02 ^{d}	3.09±0.25 ^{d}	1.91±0.01 ^{c}	8.09±0.05 ^{c}
5	0.06±0.01 ^{b}	1.13±0.05 ^{e}	2.19±0.01 ^{e}	1.82±0.01 ^{d}	6.20±0.02 ^{d}

Data are reported as means ± SD (n = 4); Different superscript letters a, b, c, d, e within colums indicate significant differences ($p<0.05$) among oil samples

Based on the data obtained for *PV*, which ranged from 1.47±0.06 to 3.09±0.25 mmol/kg (Table 1), it may be concluded that the investigated samples of cold-pressed sunflower oil are of good quality, and that 6-month storage did not cause significant increase in *PV*. However, literature also quotes findings showing significant increase in *PV* of pressed sunflower oil made from seed stored for 8 months[12].

However, statistical analysis of the *PV* data showed the existence of significant differences between the investigated oil samples. It was found that the presence of hull in pressing material (32% - Sample 2) had a positive effect on *PV* value (^{b}), causing its significant reduction. Simultaneous presence of impurities (5-10%) and hull (16-32%) (Sample 3 and 5) shows significant (^{c,e}), negative effect on *PV* increase. In the pressing material prepared from seed stored for 6 months, the maximal amount of impurities of 10% (Sample 4) showed the most significant (^{d}) and negative effect.

The *p-AnV* data for the investigated oil samples were up to 1.91±0.01 (Table 1),

which is higher in comparison to the value of cold-pressed sunflower oil made from 12-month old seed[15], but also significantly lower compared to sunflower oils obtained by extraction, for which this parameter is in the range from 1.46[16] to 4.99[17].

Data from the literature show that increase of seed storage time causes decrease of *IP* values in cold-pressed oil[15]. The results of this investigation also confirm this. Namely, according to the results of the Rancimat test, the *IP* values (at 100°C) of investigated oil samples (Table 2) were in the range from 9.35±0.02 to 10.88±0.03 h, which is significantly higher compared to the results of Dimić et al. (2004)[15] for cold-pressed sunflower oil made from 12-month old seed (7.0 h).

By comparing the *IP* values of investigated oil samples it can be concluded that the presence of impurities pressing material, differently from simultaneous presence of impurities and hull and presence of hull alone, had an adverse effect on the oxidative stability of sunflower oils made from cold-pressing of stored seed. The negative effect of impurities on oxidative stability of analyzed oils, which was identified, was also expected, because that was also concluded while producing cold-pressed oil from fresh seed[18].

The shortest induction period of 9.35±0.02 h was measured for Sample 4 with 10% impurities, while the longest *IP* of 10.88±0.03 belonged to Sample 2, which was obtained by pressing material containing no impurities and 32% of hull. The observed significantly favorable effect of hull on stability of cold-pressed sunflower oil is in agreement with the literature findings[19].

Table 2. Stability of cold-pressed sunflower oil

Sample	Rancimat test IP (h)	Schaal – test		
		PV* (mmol/kg)	CPV x 10^2 (%)	Totox*
1	9.85±0.08 ^{b}	30.10±0.22 ^{b}	18.17	59.99±0.13 ^{a}
2	10.88±0.03 ^{a}	24.47±0.19 ^{a}	15.65	49.56±0.37 ^{b}
3	10.84±0.04 ^{a}	24.69±0.87 ^{a}	11.66	50.37±1.73 ^{b}
4	9.35±0.02 ^{b}	31.06±0.29 ^{b}	9.05	64.58±0.57 ^{d}
5	9.93±0.06 ^{c}	22.84±0.03 ^{c}	9.43	47.31±0.06 ^{c}

*Values determined after holding the oil at 63±2°C for 96 h; Different superscript letters a, b, c, d within columns indicate significant differences ($p<0.05$) among oil samples

According to data found in literature, the oxidative stability of oil samples increased with decrease in the content of free fatty acids and primary oxidation products[20]. However, obtained values of the coefficients of correlation between the *IP* and *AV* (R^2= 0.4142) and between *IP* and *PV* (R^2= 0.49) (Table 1 and 2) had lower values than expected.

The results of the Schaal test (Table 2) indicate that the tempering of the oils at 63±2°C for 96 h caused a significant increase in the *PV*. The final peroxide values were found to be 9.05-fold to 18.17-fold higher than the initial *PV*.

It should be pointed out that the data for oxidative stability of the investigated oils obtained by the Schaal and Rancimat tests (Table 2) are in correlation ($R^2 = 0.49$).

Also, an insight into the oxidative state of oil samples is obtained based on the Totox values. Initial Totox values were in the range from 3.11±0.01 (Sample 2) to 8.09±0.05 (Sample 4) (Table 1). The Totox values after the four-day oil tempering at 63±2^{o}C ranged from 47.31±0.06 (Sample 5) to 64.58±0.57 (Sample 4) (Table 2). By comparing the Totox values before and after tempering it can be seen that investigated oils made from seed stored for 180 days maintained their good and best, i.e. bad and worst oxidative quality and after tempering, with the exception of Sample 5, which had significantly increased oxidative stability even after tempering, as well as Sample 1, with reduced oxidative stability after tempering.

CONCLUSION

It was concluded that the quality of cold-pressed sunflower oil made from seed stored for 6 months was good, and that separate, as well as simultaneous presence of impurities and hull in pressing material had a negative effect.

The results of these investigations also show that shelf life of oil, expressed through induction period of oil made from pressing of sunflower seed stored for 6 months, was good, and that presence of impurities had a negative effect, contrary to the simultaneous presence of impurities (5-10%) and hull (16-32%). The presence of hull alone, which was present in bigger amount (16-32%) in the pressing material, significantly increased oxidative stability of cold-pressed oil.

Summarizing the results of the investigation, it can be concluded that good-quality cold-pressed oil can be made also from seed stored for 6 months under adequate and controlled conditions. Impurities of 10% present in pressing material have the most unfavourable effect on the quality of this oil.

Acknowledgements

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COAGULATION AND FLOCCULATION IN TECHNOLOGY OF DRINKING WATER TRETMENT

PROFESSIONAL PAPER

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ABSTRACT

Recent scientific findings related to the efficiency of coagulation and flocculation provide new opportunities but also impose stringent new requirements and constraints related to water quality and processes of drinking water treatment. This imposes a continuous monitoring strategy and implementation of new information, trends and opportunities in order to increase the efficiency of these processes. The paper emphasizes the importance of proper selecting the operational parameters - type and dose of chemicals, and manipulation with chemicals. It is shown methods of selecting the type and dose of chemicals and recent findings regarding the conditions of their interference in the conventional technology of drinking water preparation within public water supply. Providing and increasing the efficiency of coagulation and flocculation means the protection of human health (through the consumption of safe drinking water), environmental protection (production of sediment and water less burdened by these chemicals) and financial savings (reduced costs in procurement of chemicals).

Keywords: coagulation, flocculation, turbidity, drinking water treatment.

INTRODUCTION

Conventional technology for drinking water treatment involves physical and chemical treatment of raw water at the plant, i.e. the use of rapid-depth filters. In raw water treatment at the plant or at facilities preceding the filter (coagulator, flocculator and sedimentation tank), water should take the allowed load that filter unit will overcome without problems, i.e. efficiently. The allowed load is 8 to 12 mg/l of suspended i.e. colloidal substances, or from 1,9 to 2,9 NTU[1]. According to recent international guidelines this allowed load is up to 1,0 (if 95% of annual raw water samples is with turbidity ≤ 10 NTU) and up to 2,0 NTU (if 95% of annual raw water samples is with turbidity > 10NTU)[2]. Regarding these limitations, it should also mention the turbidity limit, prescribed by drinking water quality standards that is 1,0 NTU for the filtered water, and the modern practice is trying to reach the turbidity of 0,1 NTU[2,3]. A prerequisite to achieve low turbidity values is efficient chemical processing, i.e. coagulation and flocculation.

EFFICIENCY FACTORS OF CHEMICALS

The aim of chemical treatment or raw water treatment with coagulants is to reduce the zeta potential of colloidal particles, allowing the particles to be enabled for interconnection as well as sticking to sand grains of the filter filling (Figure 1)[4].

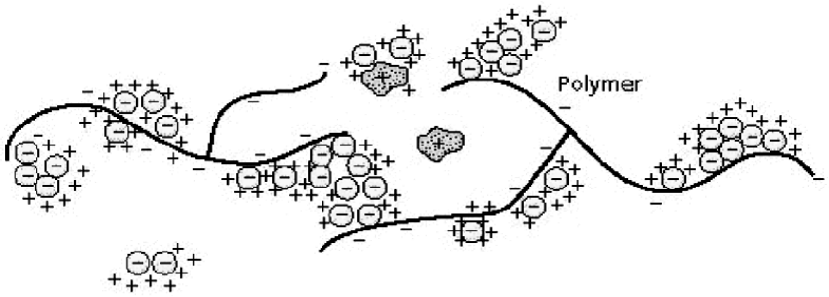

Figure 1. Influence of coagulants and flocculants to colloids[5]

Factors affecting the efficiency of coagulation and flocculation, regarding water quality parameters, are turbidity, pH and alkalinity, ionic stability and temperature[6]. Regarding the operational parameters, a major impact has intensity and duration of mixing these chemicals, their type and dose. In the operations of manipulation with the coagulant, the problems that are commonly encountered at the plant refer to:

- accuracy of adding the appropriate dose or concentration and
- provision of full and immediate coagulant dispersion.

For example, using aluminum sulfate (ALUM), as a coagulant, or polyacrylamide (PAA) as a flocculant, it is possible that some of these chemicals get into the drinking water in impermissible concentrations (MDK for PAA=0,01mg/l, MDK for Al=0,20 mg/l)[3,7]. Overuse of these chemicals is guided by the desire to achieve a lower turbidity level, which is a good goal, but high doses are not a priority method. Low turbidity is better to achieve by adequate dosing, i.e. proper manipulation of chemicals. Negativity of using large amounts of flocculants can be also very dangerous for health. Laboratory studies showed that acrylamide is a cumulative neurotoxin, and in some countries the use of this flocculant is prohibited in the technology of drinking water preparation. In Bosnia and Herzegovina, this chemical is still in use unfortunately (probably due to low price or usual practice)[6]. This imposes extra caution and the need for proper manipulation with these chemicals. Relating to these problems, the following considers exactly the selection of chemicals type and dosage and chemical handling, like the efficiency factors of coagulation and flocculation process.

DETERMINATION OF CHEMICALS TYPE AND DOSAGE

To minimize the negative impact and at the same time to increase the efficiency of use of coagulants and flocculants it should take into account of their type and dose, and in the function of the raw water composition. Below are listed and explained some of the ways of selecting the type and dose of chemicals.

Diagram of coagulation

Diagram of coagulation was developed by entering data from the literature to the stability diagram for the coagulant (pC - pH)[5,8] (Figure 2). In this way, it is possible to generalize the dosage of coagulant and pH for effective destabilization with different coagulation mechanisms. These

diagrams offer an insight into the effectiveness of various coagulants in their different doses and different pH values. Figure 2 shows the coagulation diagram of most commonly used coagulants, Al and Fe salts, or aluminum sulfate and iron chloride.

The diagram indicates the area covered by coagulation (hatch), i.e. an area of hydroxide stability ($Al(OH)_3$ and $Fe(OH)_3$). Formation of the coagulation diagram allows the prediction of favorable conditions for the operation of the filter plant.

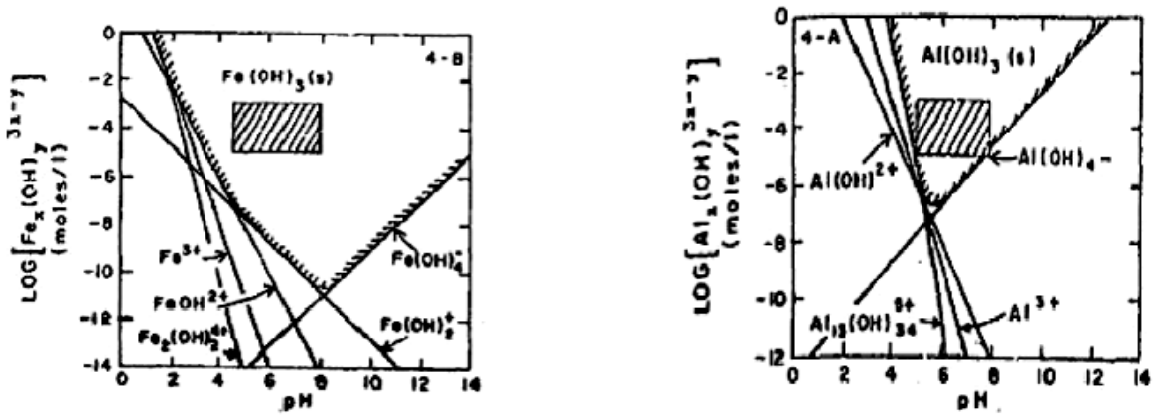

Figure 2. Coagulation Diagram [5]

The "jar" test

The optimum conditions for coagulation and flocculation can be relatively easily determined by experiment, with so called "jar" test, under conditions that simulate the real ones[4]. The "jar" test can be used in decision making, when selecting the coagulant, flocculant, their combination, sequence and the most efficient and economical dosing. These tests measure physical indicator of water quality, such as remaining turbidity and water color, in the function of the impact of coagulant type and dose, pH, Reynolds number or temperature of solution.

Empirical formulas, diagrams and tables

Determining the coagulant type and dose using the "jar" tests or using coagulation diagrams is rarely used at the plant. More often are used less reliable, but still useful, methods of determining the dose of chemicals in accordance with the change in turbidity, and possibly pH value i.e. raw water alkalinity. For this purpose, we use some appropriate formulas, diagrams or tables[6,7]. It should be noted that these methods of determining the dose of chemicals are often formed precisely on the basis of the "jar" test, which is acceptable, but requires occasional checking and possible correction during the plant operation.

WORKING WITH CHEMICALS – INTENSITY AND TIME OF MIXING

Besides the proper choice of type and dose of chemicals, it is also important to work correctly with chemicals, i.e. the way of entering and dispersion. The main problem in most of the coagulation and flocculation systems is that are designed without good

information about the optimal time (*t*) and velocity gradient, i.e. the energy required for mixing (*G*)[9]. According to data obtained from exploitation of the plants where are used the coagulation and flocculation, the necessary value of *G x t*, should be in the range of 2 x 10^4 to 2 x 10^5. Experimental studies will provide the most accurate indicators for the values of these design parameters (t) and (G).

Coagulation

According to the conventional design criteria, depending on the selected input mode of energy, the required velocity gradient (*G*) for fast mixing ranges 700-4000 s^{-1}, with the mixing time (t) from 10 to 60 s, and even a few minutes[2,10] (Table 1).

Table 1: Criteria values ((G) and (t)) for effective mixing[10]

t, seconds	G, s^{-1}
<10	4000–1500
10–20	1500–950
20–30	950–850
30–40	850–750
40–130	750–700

New theoretical and experimental studies do not use the parameter (G) in the design. According to the statement of Professor Amirtharajah, the use of this parameter in the coagulation is inadequate[6,8] The basic design parameter for mixing is time (*t*). The initial reaction of raw water with the coagulant is extremely fast and ends up in a split second. Therefore, it is essential that all raw water and coagulant are mixed in less than 1 second or before completion of the initial reaction. By this approach, use of the term the current mixing is more adequate than the term of rapid mixing. Access to the current mixing means not only savings in operating costs (more energy is used by applying (*G*) parameter),but also more efficient use of chemicals.

Flocculation

Use of flocculants is part of raw water chemical treatment. This improves the efficiency of chemical precipitation, i.e. filtration, but also reduces the required dose of coagulant. Sometimes it can omit the use of these chemicals, and with well-selected operational parameters ((*G)* and (*t)*) can be achieved favorable conditions for the formation of floccules and their efficient deposition. The dimensions of these floccules should be adjusted to the granulation of filter filling, and the strength to the filtration velocity. A well designed flocculator, with appropriate regulated speed of mixing (G), with the required retention time (t), will provide floccules of adequate seed size and strength. In the absence of experimental studies, in the flocculator design, as design parameter is used the product *G x t*. Large (*G*) and small (*t*) gives a small, but solid floccule. Small (*G*) and large (*t*) gives a large but lightweight floccule. The size of velocity gradient (G) for flocculation ranges from 20 to 150 s^{-1}, and the mixing time (t) from 20-45 minutes[6,9,10] (Table 2). These parameters depend also on the season, i.e. temperature. Winter requires a longer mixing time and vice versa. Large and solid floccules will create a well designed flocculator with different i.e. gradually decreasing of velocity gradient.

Table 2. Criteria values ((G) and (t)) for effective flocculation[10]

Type of Raw Water	**G, s^{-1}**	**G x t** (dimensionless)
Low turbidity and colored	20 - 70	50 000 – 250 000
High turbidity	70 - 150	80 000 – 190 000

CONCLUSION

In order to ensure and enhance the effects of drinking water preparation, i.e. the effects of filtering, a prerequisite is to ensure and enhance the effects of coagulation and flocculation. By properly selecting the type and dose, and choosing the appropriate velocity gradient and time of mixing chemicals, it achieve favorable chemical conditions for the construction / formation of floccules and further their deposition and retention in the filters. Achieving favorable chemical conditions means the addition of appropriate chemical in appropriate doses and with the corresponding velocity gradient and mixing time. Recent scientific findings offer the possibility of increasing the efficiency of these processes. Stricter legislation or restrictions impose the continuous monitoring strategy and application of new knowledge into practice in order to effectively use the effects of chemistry, and avoid the negative consequences of the use of chemicals. In doing so, the goal is primarily to protect public health, reducing the negative environmental impact and financial savings. All this assumes the existence of a trained and responsible personnel and professional management of these processes, adequate equipment and laboratory for monitoring and control of operational and quality parameters in the drinking water preparation.

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BENEFITS OF THERMAL TREATMENT OF ANIMAL WASTE IN THE PLANT ADVANCED TRI€CO TECHNOLOGY®

J. Hrnjica Bajramović

Grizelj

The quality of our environment doesn't depend only on regulations provided by the government and utility companies, but it also depends on the habitants of the environment, people. While living on a certain territory, you must pay a close attention to processes, in which various types and quantities of biological waste are created. A responsible and obligatory relationship toward the waste and improvement of the culture of proper waste treatment is a necessity of creating a healthier environment as the substruction of every society.

In primary and final production of animal origin, enormous quantities of biological waste were created. By-products of animal orgin are usually created during the slaughter of animals for human consumption. These products occur during the creation of animal origin products, during the treatment of dead animals and the ones killed during certain disease control measures... This waste represents a potential life hazard for people and animals, and it also affects the environment. It needs to be under control at all costs. In average, during the humane slaughter of animals different kinds of waste are created for a different species: oxen (35%), cows (30%), calves (30%), sheep (40%), poultry (35%).

According to the regulation of waste categories with list (Sl. novine Federacije BiH,number 33/03), animal waste is marked with number 02 02 – as waste that originated from preparation and processing of meat, fish, and other food of animal origin. The whole series of regulations are governing the proper treatment of this kind of biological waste, which is actually a resource that brings a considerable benefit through a safe and regular treatment. In the Decision on by-products of animal origin and their products which are not designed for human consumption (Sl. glasnik BiH, number 19/11) a categorization has been made, and the waste should be handled in accordance with it, as it reflects the legislations EU No. 1069/09 and 142/11.

By categorizing the animal waste, the one from the first category needs to be dealed with the proper treatment, while the ones from the second and third category can be treated for further usage, and therefore the Regulation on establishing animal health conditions for storage, usage, collection, transportation, identification and traceability, registration and approval of the operation, marketing, import, export and transit of by-products of animal origin and their products are not intended for human nutrition (Sl. glasnik BiH, number 30/12).

Proper disposal of all three categories of animal waste is carried out through a series of procedures that should make the treatment environmentally more

appropriate, veterinary – health safe and sanitary correct, and certainly energy efficient, which will contribute to economic profitability. In regard to the structure and properties of animal waste, the collection of the waste should be done in an appropriate way, enabling the selection, classification, marking, adequate transportation, storage and processing of waste with the realization of benefits.

Animal waste, when it just has been formed, before transport and treatment, should be kept in containers with cooling system, at temperature of + 4 ºC, and it is selected by categories and marked, and protected from contact with insects, rodents and other animals.

For easier identification, with the Regulation it has been indicated that colours should be used to mark different kinds of animal waste or animal origin products. Regulations on the surface or section of the packaging, container or a vehicle, or on a label or symbol, which are indicated on the following way:

- In the case of Category I material, BLACK colour is used.
- In the case of Category II material (other than manure and digestive tract content), YELLOW colour is used.
- In the case of Category III materials, GREEN colour with a high concentration of BLUE is used to ensure that it's clearly recognizable in comparison to other colours.

For a better identification of the packaging, container, or vehicle, following things must be clearly and legibly written:

- In the case of Category III material: "it's not intended for human consumption".
- In the case of Category II material (other than manure and digestive tract content) and products derived from animal origin by-products from Category II material: "not intended for feeding animals," unless the material from Category II is intended for feeding animals (zoo animals, reptiles and birds of prey (that are not in the zoo), furred birds, wild animals, dogs from registered farms, breeding of hunting dogs, dogs and cats in shelters, labels, instead of "feeding ..." will be complemented with a special name for the species for which the feeding material had been intended in the first place.
- In the case of Category I material and products obtained from materials of Category I, if they are intended for disposal "for disposal" or "only for the manufacture of products obtained from products of animal origin by-products. Not intended for human or animal nutrition, nor for usage on land“.
- In the case of manure and digestive tract content: "manure".

A place of treatment of the waste is always a certain distance away from the place where it was originally created, as well as one of the most critical point of disposal of animal origin waste is the transportation. It must be performed in a prescribed manner in sealed new packaging or covered leakproof containers or in vehicles. Vehicles and containers are maintained in clean condition, cleaned, washed and disinfected and dry to avoid cross contamination. Containers for multiple usage can be used, if the authorised veterinarian approved such

usage. Transportation must be provided without violating public safety and to prevent any possible form of contamination.

In order to avoid any risks to human and animal health, animal waste products derived from it must be kept at the regulated temperature. Unprocessed category III material is intended for animal food or raw pet food production, and it must be stored and transpored, when it's cooled at + 4 ºC, frozen or ensiled, unless there had been types of processing within 24 h after collection, or after the storaging finishes in cooled or frozen state, if no additional transportation is done by means of transport which maintains the temperature of storage. The animal waste should be disposed immediately or no later than 12 hours after it has been created in summer, 24 hours in winter, while in other seasons never later than 48 hours.

Animal waste after collection, selection, marking and transport must be properly disposed of in order to complete the treatment in safe and fair manner. As the categorization of animal waste is done with a certain level of risk, it is prescribed precisely how to handle and set the treatment of the same animal waste.

Facilities/Plants for the treatment of animal waste should be affordable for continuous cleaning, washing and disinfection, the well-drained hard surface. Staff working in those plants must have a sufficient number of toilets with wash basins and showers, a changing room, a rest room and a workshop, in order to prevent any form of possible contamination. If the facility/plant is located directly on the farm there must be a physical separation, if necessary, fences and highly professional equipment. Plants intended for incineration must be designed, built and equipped to work in a way that waste is treated at temperatures over 860 °C with a co-incineration at a temperature of 1200 °C with retention of at least 2 ". The second and third categories can be sterilized at temperatures over 130 °C, with a pressure of 3 bar and uninterrupted period of at least 20 minutes, and the particles dispersed at 50 mm should be treated to the final product. Techniques must be used to monitor the parameters and conditions that are relevant to the process of incineration and co-incineration. Plants in which by-products of animal origin are treated and their products with a capacity exceeding 50kg/h must be equipped with at least one auxiliary burner that can turn itself on when the temperature of combustion gases, after the last injection system of combustion air falls below 850 °C or 1100 °C, as it should be. Plants must be operated in a such way that by-products of animal origin remain only eco ash that can not be packed chilled and can be used as an inert material for the embankments.

If there are sufficient amounts of animal waste, and analysis in Bosnia have shown that enormous quantities, and types of waste treatment in a stationary plant eco Advanced TRI€CO Technology® of 24 h / 365 days are possible to be done with significant benefits. After the selection, according to prescribed sterilization conditions at the plant Advanced TRI€CO Technology®, a category II material and category III material, the production of ecological food is enabled: blood, bone and meat meal, and eco fats and oils and eco-fertilizer, and with incineration of category I material an eco ash is obtained. Organic food is used for pet and zoo animals food, while fats and oils

are used as a resource in the cosmetic industry. Eco-fertilizer has the quality and is provided for usage in horticulture, sports fields and eco ash can be used for various levees. Benefit of animal waste disposal is representative even through production of energy. With processes of cogeneration (CHP), trigeneration (CHCP) and polygeneration, obtained energy can be thermal, cooling and electric, depending on the amount provided for the treatment and opportunities for placement of the very same energy. The authorised veterinarian supervises the fulfillment of conditions for the entire chain of collection, selection, marking, transportation and treatment, as well as final products.

It is time to consider the waste as a chance for benefit, with responsobility and obligations, that are prescribed to us with legislative framework.

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TECHNOLOGICAL NOTES

New cultures remove cheese bitterness, Chr Hansen

Chr Hansen is launchinga new culture range wich it says can remove bitterness in cheese.

Althought the ingredients firm has previously launched cultures to improve the flavour profile of food products, the new Mild O range is specifically targeted for the cheese segment, Jamila Bouanda, a spokesperson for the company told DairyReported.com.

The new cultures, wich some in both freeze – dried and frozen formats, can be used in different types of cheesea, soft cheese, white brined cheese, quark and gourment style continental cheeses made from milk blends, said Bouanda.

Acidification process

The mesofilic strain that are in the Mild O blends have been carefully selected among several hundred strain explained the spokesperson, adding that the primary reason for their mild flavour profile is the hight content of 'cremoris' subspecies strain.

The culture blends show down the acidification process wich removes the risk of a brittle or hard cheese texture, said the firm. In addition to being salt sensitive, it added.

˝This is an adventage to cheese producers who wish to avoid post acidification usually causing sour flavour notes in the cheese,˝ said Theis Bacher, marketin menager, Cheese Cultures, Chr Hansen.

˝Using a 'Mild O' cultures blend, producers can simply stop the acidification process at the high pH and thereby avoid excessive sourness.˝

Other benefits

As well as the culture's bitterness reduction, the range can offer other functions, said Bounada, according to the type of cheese.

These include a reduction in fermentation time, improved cheese flavour, the avoidance of flotation curd and an increased robustness system, she said.

Mild O is expected to gain solid footing in several markets and cheese segments within the next couple of years due to their *˝powerful mildness profile and versatile application˝,* according to Chr Hansen.

Introductory trials have shown that there is particularly great potential in specific Southern European gourmet cheese segments such as manchego and pecorino sardo, said the firm.

Artificial Photosynthesis Breakthrough: Fast Molecular Catalyzer

ScienceDaily Researchers from the Department of Chemistry at the Royal Institute of Technology (KTH) in Stockholm, Sweden, have managed to construct a molecular catalyzer that can oxidize water to oxygen very rapidly. In fact, these KTH scientists are the first to reach speeds approximating those is nature's own photosynthesis. The research findings play a critical role for the future use of solar energy and other renewable energy

Researchers all over the world, including the US, Japan, and the EU, have been working for more than 30 years on refining an artificial form of photosynthesis. The results have varied, but researchers had not yet succeeded in creating a sufficiently rapid solar-driven catalyzer for oxidizing water.

"Speed has been the main problem, the bottleneck, when it comes to creating perfect artificial photosynthesis," says Licheng Sun, professor of organic chemistry at KTH.

But now, together with research colleagues, he has imitated natural photosynthesis and created a record-fast molecular catalyzer. The speed with which natural photosynthesis occurs is about 100 to 400 turnovers per seconds. The KTH have now reached over 300 turnovers per seconds with their artificial photosynthesis.

"This is clearly a world record, and a breakthrough regarding a molecular catalyzer in artificial photosynthesis," says Licheng Sun.

The fact that the KTH researchers are now close to nature's own photosynthesis regarding speed opens up many new possibilities, especially for renewable energy sources.

"This speed makes it possible in the future to create large-scale facilities for producing hydrogen in the Sahara, where there's an abundance of sunshine. Or to attain much more efficient solar energy conversion to electricity, combining this with traditional solar cells, than is possible today," says Licheng Sun.

He points to the problem of skyrocketing gasoline prices, and these advances with the rapid molecular catalyzers can in turn lay the groundwork for many important changes. They make it possible to use sunlight to convert carbon dioxide into various fuels, such as methanol. And, technology can be created to convert solar energy directly into hydrogen. Licheng Sun adds that he and his research colleagues are working hard and pursing intensive research to make this technology reasonably inexpensive.

"I'm convinced that it will be possible in ten years to produce technology based on this type of research that is sufficiently cheap to compete with carbon-based fuels. This explains why Barack Obama is investing billions of dollars in this type of research," says Licheng Sun.

He has conducted research in this field for nearly twenty years, more than half of that time at KTH, and adds that he and many other researchers see efficient catalyzers for oxidation of water as key to solving the solar energy problem.

"When it comes to renewable energy sources, using the sun is one of the best ways to go," says Sun.

The research findings are of such importance that they have recently attracted the attention of the scientific journal *Nature Chemistry*.

The research pursued by Licheng Sun and his colleagues is funded by the Wallenberg Foundation and the Swedish Energy Agency. They collaborate with researchers at Uppsala University and Stockholm University, and, together with Professor Lars Kloo at KTH, they run a joint research center involving KTH and Dalian University of Technology (DUT) in China.

Algae Biofuels: The Wave of the Future

ScienceDaily Researchers at Virginia Bioinformatics Institute have assembled the draft genome of a marine algae sequence to aid scientists across the US in a project that aims to discover the best algae species for producing

The results have been published in*Nature Communications.*

The necessity of developing alternative, renewable fuel sources to prevent a potential energy crisis and alleviate greenhouse gas production has long been

recognized. Various sources have been tried -- corn for ethanol and soybeans for biodiesel, for example. But to truly meet the world's fuel needs, researchers must come up with a way to produce as much biofuel as possible in the smallest amount of space using the least amount of resources.

Enter algae. Unlike other crops like corn or soybeans, algae can use various water sources ranging from wastewater to brackish water and be grown in small, intensive plots on denuded land. While algae may still produce some $C0^2$ when burned, it can sequester $C0^2$ during growth in a way that fossil-fuel based energy sources obviously can't.

Scientists in VBI's Data Analysis Core (DAC), Robert Settlage, Ph.D., and Hongseok Tae, Ph.D., assisted in the assembly of the genome of *Nannochloropis gaditana*, a marine algae that may be capable of producing the lipid yields necessary for a viable fuel source.

"Getting the data is now the easy part. What we're doing in the DAC is enabling researchers to move beyond informatics issues of assembly and analysis to regain their focus on the biological implications of their research," said Settlage.

Further analysis revealed that with fairly straightforward genetic modification, *N. gaditana* should be capable of producing biofuel on an industrial scale, which may be the wave of the future in fuel research and production.

MOFs for natural gas purification

The iron-MOF is also good at purifying natural gas, which is a mixture of methane and various types of hydrocarbon impurities that have to be removed before the gas can be used by consumers. These impurities can then be sold for other uses, Long said.

"MOF compounds have a very high surface area, which provides lots of area a gas mixture can interact with, and that surface contains iron atoms that can bind the unsaturated hydrocarbons," Long said. "Acetylene, ethylene and propylene will stick to those iron sites much more strongly than will ethane, propane or methane. That is the basis for the separation."

Nickias noted that increased supplies of natural gas from shale have provided more opportunity to extract and use ethylene and propylene from natural gas, and a variety of materials and approaches are being examined to cut energy use during the refining and purification of olefins.

"Significant energy savings could be achieved if a non-distillation separation could be implemented, or more realistically, the load on a cryogenic distillation unit can be reduced via upstream modifications to the process," Nickias said.

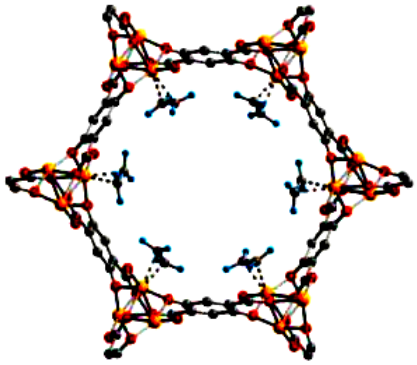
Petroleum refined for the chemical industry is typically a mix of hydrocarbons, primarily two-carbon molecules -- ethane, ethylene and acetylene -- and three-carbon chains -- propane and propylene. Cryogenic distillation separates these compounds -- all of them gases at room temperature -- by liquefying them at low temperatures and high pressure, which causes them to separate by density. Ethylene and propylene go into plastic polymers, while ethane and propane are typically used for fuel.

The researchers found that when pumping a gas mixture through the iron-based MOF (Fe-MOF-74), the propylene and ethylene bind to the iron embedded in the matrix, letting pure propane and ethane through. In their trials, the ethane coming out was 99.0

to 99.5 percent pure. The propane output was close to 100 percent pure, since no propylene could be detected.

After the ethane and propane emerge, the MOF can be heated or depressurized to release ethylene and propylene pure enough for making polymers.

"Once you saturate the material - with ethylene, for example -- you shut off the valve, stop the feed gas, warm up the absorber unit and the ethylene would come out in pure form as a gas," Long said.



This is a cross-section of the iron-based MOF bound to six ethylene molecules, as determined by neutron diffraction. The MOF consists of a carbon (gray) and oxygen (red-orange) framework with iron atoms (yellow-orange) at strategic sites to bind the ethylene carbon atoms. The attraction between the iron and unsaturated hydrocarbons like ethylene (olefins) allows the MOF to adsorb these hydrocarbons, separating them from saturated hydrocarbons (paraffins) even at high temperatures. (Credit: NIST and Jeffrey Long lab, UC Berkeley)

MOFs are like packed soda straws

Through a microscope, Fe-MOF-74 looks like a collection of narrow tubes packed together like drinking straws in a box. Each tube is made of organic materials and six long strips of iron, which run lengthwise along the tube. Analysis by Long's colleagues at the NIST Center for Neutron Research showed that different light hydrocarbons have varied levels of attraction to the tubes' iron. By passing a mixed-hydrocarbon gas through a series of filters made of the tubes, the hydrocarbon with the strongest affinity can be removed in the first filter layer, the next strongest in the second layer, and so forth.

"It works well at 45 degrees Celsius, which is closer to the temperature of hydrocarbons at some points in the distillation process," said coauthor Wendy Queen, a postdoctoral fellow at NIST who worked for six months in Long's UC Berkeley lab. "The upshot is that if we can bring the MOF to market as a filtration device, the energy-intensive cooling step potentially can be eliminated. We are now trying out metals other than iron in the MOF in case we can find one that works even better."

Long and his laboratory colleagues are developing iron-based MOFs to capture carbon from smokestack emissions and sequester it to prevent its release into the atmosphere as a greenhouse gas. Similar MOFs, which can be made with different pore sizes and metals, turn out to be ideal for separating different types of hydrocarbons and for storing hydrogen and methane for use as fuel.

Long's other colleagues are UC Berkeley graduate students Eric D. Bloch and Joseph M. Zadrozny; Rajamani Krishna of the Van't Hoff Institute for Molecular Sciences at the University of Amsterdam; and Craig M. Brown of NIST and The Bragg Institute at the Australian Nuclear Science and Technology Organisation in Menai, New South Wales.

The research is part of the Center for Gas Separations Relevant to Clean Energy

Technologies, an Energy Frontier Research Center funded by the Department of Energy that focuses primarily on creating novel materials for capturing and storing carbon dioxide.

Fool's Gold may prove an unlikely alternative to overexploited catalytic materials

Catalytic materials, which lower the energy barriers for chemical reactions, are used in everything from the commercial production of chemicals to catalytic converters in car engines. However, with current catalytic materials becoming increasingly expensive, scientists are exploring viable alternatives.

Researchers at the University of Cambridge have now discovered that the sulphide material iron pyrite, commonly known as „Fool's Gold“, may hold the answer. Their findings were published online, 10 February 2012, in Physical Chemistry Chemical Physics.

In the past, sulphur was believed to be one of the most detrimental elements for surface reactions, able to decrease dramatically the reactivity of a catalyst by occupying (poisoning) the „active sites“ on the material, but more recently some sulphur materials (for example, molybdenum sulphides) have actually shown interesting catalytic properties of their own.

Using state – of – the – art electronic structure calculations, researchers led by Stephen Jenkins at the University's Department of Chemistry, explored the potential catalytic activity of iron pyrite, the most abundant sulphur mineral on Earth. In their study, the Cambridge researchers focused on the reactions between iron pyrite and nitrogen oxides (NO_x), an extremely poisonous class of compounds produced (among other sources) by car engines and industrial power plants.

Dr Marco Sacchi, the first author on the paper, said: „Recent European legislation has proposed increasingly strict legislative limits on the concentration of NO_x that can be emitted by vehicles; therefore the search for new and more efficient catalysts that can capture these molecules and transform them into innocuous gases such as nitrogen and water vapour, is urgently relevant.“

Developing new catalysts derived from inexpensive minerals, instead of increasingly costly (and rare) precious metals, is an important area of research that involves several groups around the world. The next steps for the Cambridge researchers will be to investigate the activity of pyrite surfaces for strategically important industrial reactions, such as the manufacture of ammonia for fertilisers, the production of synthetic hydrocarbon fuels from renewable biomass, and the extraction of hydrogen for use in the future fuel cell electric vehicles.

Dr Sacchi added: „The necessity of finding reliable alternative to overexploited catalytic materials – such as platinum, rhodium and gold – will soon become unavoidable. Experimental work is currently underway in our group, and we hope that our work will ultimately allow us to test the potential for catalytic application of a wide range of sulphidic and carbidic materials. In future, we aim to develop fruitful scientific collaborations with chemical engineering groups and with industrial partners. “

http://www.cam.ac.uk/research/news/a-mineral-way-to-catalysis/

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