

# THE CHEMICAL COMPOSITION ASSESSMENT OF THE FETEASCĂ NEAGRĂ GRAPE POMACE AND ITS FRACTIONS OBTAINED FROM WINE INDUSTRY IN DIFFERENT YEARS

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**Keywords:** grape pomace, fractions, chemical content, polyphenols, tannins.

**Abstract:** The aim of the hereby study was to analyze and to compare the chemical content of the grape pomace and its fractions: skins and seeds from the red grape variety Fetească neagră (from Iași area), obtained in different years 2013 and 2014 respectively, from the winemaking process. Measurements targeted the dry matter content (DM%), organic matter (OM%), crude ash (CA%), crude protein (CP%), crude fat (EE%), crude fiber (CF%), nitrogen-free extractive substances (NFES%), total polyphenols (TP%) and tannins (Ta%). The results obtained showed significant differences in the chemical composition in favour of the grape pomace obtained in the 2014 climatic conditions: in the case of the seed for the content of DM%, SEN%, TP% and Ta%, in the case of the skins for the content of DM%, OM%, CF%, TP%, Ta% and in the case of the grape pomace for the content of DM%, OM%, CF%, SEN%, TP%, and Ta%. Comparative analysis of the chemical composition showed an annual variation of the chemical components, which may be due to climatic conditions and winemaking process. Therefore, an annual chemical quality assessment of the grape pomace is necessary, for the efficient use in the animal feed.

## INTRODUCTION

The grape pomace is a by-product generated during the winemaking process, in a very large amounts which contains a variable proportion of seeds, skins and stalks fragments.

Studies on the chemical composition of the grape pomace revealed that in addition to its content in proteins, fats, sugars and minerals (Pop I.M., *et al.*, 2014; Baumgartel T., 2007; Zalikarenab L.R., *et al.*, 2007) it also contains high amounts of polyphenols with antioxidant properties (Alonso A., *et al.*, 2002). Polyphenols accumulate in the solid parts of the grapes, in skins (28-35%) seeds (60-70%) and stalks (less than 10%) which pass, after the maceration-fermentation process into the grape juice and wine, up to 50% of them, the rest remaining in the grape pomace (Bișboacă S.E., 2012).

The skins from the red grapes are rich in anthocyanins, the hydroxy acids and flavonol glycosides, while the seeds contain mainly flavanols and gallic acid. Flavonoids are distributed in the seeds and stalks that mainly contain catechins, epicatechin and procyanidins (Xia E.Q., *et al.*, 2010).

Several studies have shown the beneficial effects of polyphenols including the antioxidant, antibacterial, anti-inflammatory and anti-methanogenic ones, as well as a protective role against the degenerative diseases thus being able to present a great interest in animal health and nutrition (Brenes A., *et al.*, 2008).

Various studies indicate that the differences in the grape pomace chemical composition are due to both the grape variety and species and to their growing conditions (Rondeau P., *et al.*, 2013; Zalikarenab L.R., *et al.*, 2007), but also due to the way the grape pomace is dried and stored (Pirmohammadi R., *et al.*, 2007).

The objective of the hereby study was the comparative assessment of the chemical content of the grape pomace and its fractions: skins and seeds from the red grape variety Fetească neagră, obtained in different years 2013 and 2014 respectively, from the winemaking process.

## MATERIALS AND METHODS

The fresh grape pomace (FGP) resulted from the red (Fetească neagră of Iași area) vinification process was naturally dried, in a clean and well ventilated space at a constant temperature of 20°C. Following the drying process, the vegetal fractions (seeds, skins) from the red grape pomace were separated to determine the chemical content of each fraction in part.

In order to determine the chemical composition, the samples were milled up to particles of 1 mm diameter, according to the standard (SR ISO 6498:2001). The chemical composition intended to determine the dry matter (DM%), crude ash (CA%), crude protein (CP%), crude fat (EE%), crude fibre (CF%) using standardized methods (SR EN ISO 5983-1:2006/AC:2009; SR EN ISO 2171:2010; SR EN ISO 6865:2002; SR ISO 6496:2001; SR ISO 6492:2001); total polyphenols (TP%) and tannins (Ta%) of the alcohol extracts (50%) of the pomace obtained from the grape pomace samples according to the patent (Bișboacă S.E., 2012) were determined by the Folin-Ciocalteu method (Singleton and Rossi, 1965). Determination of chemical components (DM%, Ash%, CP%, EE%, CF%) helped to obtaining through mathematical

calculation of organic matter (OM%) and nitrogen-free extractive substances (NFES%). Three determinations were made for each sample of fraction and grape pomace analysed.

Means and standard deviations were calculated for each parameter. The expression of the chemical composition of the analysed grape pomace and grape pomace fractions was made by reporting all the compounds analysed to the DM. The results obtained were statistically processed using the single factor ANOVA test ( $p < 0.05$ ) to highlight significant differences between the calculated values.

## RESULTS AND DISCUSSIONS

Comparative analysis of the chemical components of seeds from the red grape pomace of the Fetească neagră obtained in different years, 2013 and 2014 respectively, is shown in the table 1.

**Table 1 Chemical composition of seeds**

Parameter		Results (Mean $\pm$ SD)		Statistical significance (Anova)	
		Seeds-2013	Seeds- 2014	p-value	Significance threshold
DM		93.09 $\pm$ 0.04	94.25 $\pm$ 0.006	9.80E-07	$p < 0.001$
% of DM	CA	3.08 $\pm$ 0.16	3.27 $\pm$ 0.06	0.11	$p > 0.05$
	OM	96.92 $\pm$ 0.16	96.53 $\pm$ 0.06	0.014	$p < 0.05$
	CP	14.47 $\pm$ 0.25	11.38 $\pm$ 0.31	0.0002	$p < 0.001$
	EE	14.82 $\pm$ 0.6	13.21 $\pm$ 0.07	0.0097	$p < 0.01$
	CF	45.57 $\pm$ 1.62	38.19 $\pm$ 0.42	0.0016	$p < 0.01$
	NFES	22.07 $\pm$ 1.54	33.74 $\pm$ 0.56	0.00025	$p < 0.001$
	TP	4.22 $\pm$ 0.08	5.23 $\pm$ 0.09	5.95E-24	$p < 0.001$
	Ta	3.63 $\pm$ 0.09	4.47 $\pm$ 0.1	9.55E-21	$p < 0.001$

The results revealed that the seeds from the grape pomace obtained in 2013 had a significantly higher content of OM (96.92 $\pm$ 0.16%), CP (14.47 $\pm$ 0.25%), EE (14.82 $\pm$ 0.6%), CF (45.57 $\pm$ 1.62%), compared to the one obtained in 2014. Instead, the seeds from the grape pomace obtained in 2014 showed a significantly higher content in DM (94.25 $\pm$ 0.006%), TP (5.23 $\pm$ 0.09%), Ta (4.47 $\pm$ 0.10%) and NFES (33.74 $\pm$ 0.56%).

There is insufficient data on proteins content in grape seeds, in the published literature (Kamel S., *et al.*, 1985; Ohnishi M., *et al.*, 1990). The seeds are not considered to be an important source of protein, their average content can vary between 11-13% (Fantozzi P., 1981; Goni I., *et al.*, 2005), and it depends on the grape variety, fertilization and climate conditions.

The oil content of the seeds may vary from 11.6 to 19.6%, being dependent on the variety and maturity of the grapes (Rao P.U., *et al.*, 1994).

Seeds total polyphenols content differs depending on the grape varieties, climate and environmental conditions, soil type, degree of ripeness and processing method of grapes in winemaking process (Fuleki T., *et al.*, 1997).

Our results on the crude chemical content and seed polyphenolic compounds are comparable to those in the published literature (Kamel S., *et al.*, 1985; Ohnishi M., *et al.*, 1990, Fuleki T., *et al.*, 1997).

The differences of chemical composition of seeds from the grape pomace of the same grape variety but obtained in different years may be due to pedoclimatic conditions, grape processing technology, maceration-fermentation time on the pomace and preservation period of grape pomace.

Almost the entire fraction of soluble carbohydrates is fermented by the wine specific microbial flora, when the maceration-fermentation period is longer (Baumgartel T., *et al.*, 2007). A higher quantity of polyphenolic compounds can be extracted in wine from the solid fractions of pomace, when the maceration period is prolonged, which may lead to decreased of these compounds in the grape pomace.

In table 4 is presented the comparative analysis of the chemical composition of skins and stalks from the grapes pomace obtained in different years.

**Table 2 Chemical composition of skins**

Parameter		Results (Mean $\pm$ SD)		Statistical significance (Anova)	
		Skins + stalks (6,77%)- 2013	Skins + stalks (9,65%)- 2014	p-value	Significance threshold
DM		93.04 $\pm$ 0.01	93.21 $\pm$ 0.02	0.0001	p<0.001
% of DM	CA	11.60 $\pm$ 0.23	7.40 $\pm$ 0.21	2.06E-05	p<0.001
	OM	88.40 $\pm$ 0.23	92.60 $\pm$ 0.21	2.06E-05	p<0.001
	CP	19.08 $\pm$ 0.66	15.26 $\pm$ 0.12	0.0006	p<0.001
	EE	3.65 $\pm$ 0.22	3.36 $\pm$ 0.12	0.11	p>0.05
	CF	12.13 $\pm$ 0.09	19.31 $\pm$ 0.35	4.29E-06	p<0.001
	NFES	53.54 $\pm$ 0.72	54.67 $\pm$ 0.26	0.06	p>0.05
	TP	1.95 $\pm$ 0.05	2.92 $\pm$ 0.03	2.81E-34	p<0.001
	Ta	1.30 $\pm$ 0.01	2.36 $\pm$ 0.06	1.26E-32	p<0.001

The results obtained revealed that the seeds from the grape pomace of 2014 had a significantly higher content in DM (93.21 $\pm$ 0.02%), OM (92.60 $\pm$ 0.21%), CF (19.31 $\pm$ 0.35%), TP (2.92 $\pm$ 0.03%) and Ta (2.36 $\pm$ 0.06%), appreciation supported by the statistical calculation (p<0.001). Instead, the skins from the grape pomace obtained in 2013 showed a significantly higher content in CP (19.08 $\pm$ 0.66%), CA (11.60 $\pm$ 0.23%), compared to the one obtained in 2014.

No significant differences (p>0.05) were observed regarding the content of crude fat and nitrogen-free extractive substances, of the skins from 2013 and 2014 grape pomace.

The increase of the skins chemical components from the grape pomace obtained in 2014 may be due to a higher proportions of stalks: 9.65 g stalk from 100 g of skins and stalks compared to the one obtained in 2013, namely 6.77 g stalk from 100 g.

The differences of chemical composition of skins and stalks from the grape pomace obtained in different years may also be due to pedoclimatic conditions of those years and grape processing technology.

The comparative analysis of the chemical composition of the grape pomace obtained in different years, is presented in Table 3.

**Table 3 Chemical composition of grape pomace**

Parameter		Results (Mean $\pm$ SD)		Statistical significance (Anova)	
		Grape pomace-2013	Grape pomace-2014	p-value	Significance threshold
DM		92.30 $\pm$ 0.01	94.06 $\pm$ 0.02	2.89E-08	p<0.001
% of DM	CA	7.12 $\pm$ 0.05	5.53 $\pm$ 0.05	8.96E-06	p<0.001
	OM	92.29 $\pm$ 0.09	94.12 $\pm$ 0.05	6.87E-06	p<0.001
	CP	16.60 $\pm$ 0.12	13.55 $\pm$ 0.04	1.89E-06	p<0.001
	EE	10.19 $\pm$ 0.09	9.24 $\pm$ 0.08	0.00016	p<0.001
	CF	28.54 $\pm$ 0.52	30.27 $\pm$ 0.7	0.027	p<0.05
	NFES	36.95 $\pm$ 0.33	41.05 $\pm$ 0.64	0.00059	p<0.001
	TP	2.20 $\pm$ 0.08	4.00 $\pm$ 0.06	8.79E-06	p<0.001
	Ta	1.44 $\pm$ 0.06	3.34 $\pm$ 0.07	5.94E-06	p<0.001

The results obtained showed a significantly higher content in the case of 2014 grape pomace for the following components: DM (94.06 $\pm$ 0.02%), OM (94.22 $\pm$ 0.05%), CF (30.27 $\pm$ 0.70%), TP (4.00 $\pm$ 0.06%), Ta (3.34 $\pm$ 0.07%) and NFES (41.05 $\pm$ 0.64%). Instead, the grape pomace obtained in 2014 showed a significantly higher content in CA (7.12 $\pm$ 0.05%), CP (16.60 $\pm$ 0.12%) and EE (10.19 $\pm$ 0.09%) compared to the one obtained in 2014.

The increase of the grape pomace chemical components obtained in 2013 (CA%, CP%, EE%) may be due to higher proportions of seeds (59,47%) compared to the grape pomace obtained in 2014 which had a lower proportion of seeds (50 %).

The higher content of CF% and NFES% in the 2014 grape pomace may be due to higher proportions of skins (41%) compared to the one obtained in 2013 (33,76%).

In the case of red winemaking, the skins are crushed and crumbled by the pressing process, which can decrease the proportion of these fractions in the grape pomace biomass.

A longer contact time between juice and solid parts of the grapes lead to lower content of polyphenols, including tannins from the grape pomace (Gomez-Plaza E., *et al.*, 2006).

In addition to proportion of seeds, skins and stalks from the grape pomace biomass, the chemical composition differences between the two types of grape pomace may also be due to pedoclimatic conditions of those years, grape processing technology, fermentation - maceration time on the pomace and preservation period of grape pomace.

## CONCLUSIONS

Our research revealed a higher content of nutritional substances in the fractions of grape pomace obtained in 2014 climatic conditions: DM%, NFES%, TP% and Ta%, in the seeds, DM%, OM%, CF%, TP%, Ta% in the skins; DM%, OM%, CF%, NFES%, TP% and Ta% in the grape pomace. Comparative analysis of the chemical composition showed an annual variation of the chemical components, which may be due to climatic conditions and winemaking process. Therefore, an annual chemical quality assessment of the grape pomace is necessary, for the efficient use in the animal feed. Also, our data supports the possibility of the grape pomace use or its fractions in the farm animals' feed, in compliance with the maximum permitted levels of crude fiber for the animal category and species concerned.

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