Researches on the Chemical Composition and the Rheological Properties of Wheat and Grape Epicarp Flour Mixes

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Grape skin is considered a valuable by-product for antioxidant and antibacterial agent preparation. The grape skin contains some active compounds, such as, dietary fiber, polyphenols, flavonols, and resveratrol; it is commonly used as a nutritional supplement. The main aim of this study was to establish the optimum dose of grape skin flour, to be used as a functional ingredient in the bakery products industry, from both chemical and rheological point of view. The laboratory experiments evaluated the functional potential of wheat flour enriched with grape skin flour, in different proportions, by examining the chemical composition and rheological behaviour of the doughs. Protein, crude fibre, fat, ash and mineral contents were determined. Using ^{1}H-NMR spectral technique, the fatty acids composition was determined, especially the concentrations of short-chain saturated fatty acids (C4-C8), di-unsaturated fatty acids, mono-unsaturated fatty acids and long-chain saturated fatty acids (>C8). 1H-NMR spectra were recorded on a Bruker Ascend 400 MHz spectrometer. The rheological behaviour was analyzed using the predefined Chopin + protocol on Mixolab, an equipment of CHOPIN Technologies. Grape skin flour was incorporated into wheat flour at three different levels, 5, 10 and 15% and it was found that incorporation up to a 15% level into the formulation of wheat flour yielded an acceptable product in terms of rheological parameters, with improved chemical, nutritional and functional properties.

Keywords: bakery, crude fibre, functional ingredient, grape skin, dough, rheological properties

Vitis vinifera (common grape vine) is a species of *Vitis*, native to the Mediterranean region, central Europe, and southwestern Asia, from Morocco and Portugal north to southern Germany and east to northern Iran. There are currently between 5,000 and 10,000 varieties of *Vitis vinifera* grapes though only a few are of commercial significance for wine and table grape production.

The archaeological discoveries revealed that wine-making dated back about 7,000 years ago, in a Neolithic village located in Iran's northern Zagros Mountains, being until now the earliest evidence of wine-making [1].

Nowadays grapes are among the most valued conventional fruits in the world and 80% of the grape yield is used for wine-making. The wine-making industries produce millions of tons of residues (grape seeds and skin) after fermentation, which represents a waste management issue both ecologically and economically [2]. The productive use of such by-products could offer substantial economic and health benefits.

These by-products (grape seeds and skin) contain active compounds, such as, dietary fibre [3], antioxidants: polyphenols (flavonoids, antocianosides etc.) [4-9], which are of growing interest to researchers [10-13], because of their linkage to human health. This content varies widely, depending on many factors: varieties of the species, soil qualities and its management [14-23], weather conditions in which the plant lives: precipitation, climate, sunny days/ year, pollution in the area of culture (especially waste water and inadequate management of different types of wastes [24-29]), conditions for obtaining and preserving the finished product, technology used, etc. [30,31].

Several studies have shown that many bioactive components in grape seeds and skin have been shown to prevent a wide array of chronic disorders linked to metabolic syndrome [32]. Grape seeds and skin flour, a polyphenol rich mixture containing flavonoids, non flavonoids, oligomeric proanthocyanidins, is commonly used as a nutritional supplement [33].

Recent research has demonstrated that grape seeds catechins protect rat cortical astrocytes against palmitic acid-induced lipotoxicity [34].

Due to the abundance of natural polyphenolic substances, grape seeds are widely used for the treatment of prostate deficits [35]. It is demonstrated that grape seeds have anti-inflammatory benefits [36], scavenging free radicals, inhibiting lipid peroxidation, anti-carcinogenic and so forth [37]. Some studies found that grape seed-derived polyphenols extract (GSP) had prostatic protective nature in vivo and could modulate prostatic oxidative stress [38,39].

Because of the increased attention to sustainable of agricultural practices, there is a vast array of applications for grape pomace, such as functional food (dietary fibre and polyphenols), food processing (biosurfactants), cosmetic (grape seeds oil and antioxidants), pharmaceutical and supplements (grape pomace powder) [40-42].

The importance of knowing the grape pomace composition enables us to find the industrial uses [43] and to evaluate the importance of the raw material variability [44] on the final application. Despite a substantial number of studies using grape pomace for different applications,

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in reality they are often ineffective as they are not successfully implemented in larger scales [45].

However the use of suitable by-products (such as wine residues) as functional ingredients has the advantage that food manufacturers can add extra value to food products, but the main factors that have to be considered are the variations affecting the processing conditions, the sensory properties, and the nutritional value of the final product.

Bakery products could represent a potential candidate for the addition of this functional ingredients, but their nutritional profile has to be improved in view of formulating functional products. Several researches have been directed to reduce fats, sugars, and energy level [46-48] of this type of products, and to improve the quality of raw materials, respectively [49].

The objective of this work was to characterize mixtures of wheat flour and grape skin flour through a physico-chemical evaluation of the rheological properties, which are the main quality attributes of these mixtures for use in bakery.

Experimental part

Materials

Grapes skin flour, a by-product obtained during manufacture of red *Vitis vinifera* from Romania, was furnished by a local wine-making factory. Grape skins were collected after the grapes were crushed and the grape juice was obtained. Fresh samples were manually sieved to separate skin fraction from the seeds. Skin fraction was dried and ground. The level of degradation of the components of this material may be considered low because all the steps were performed at low temperature.

Wheat flour used in the study was 480 type (ash, d.m. – 0.48%) and was provided by Titan S.A. (Bucharest, Romania).

Preparation of wheat flour mixtures enriched in bioactive compounds

Three types of mixtures of 650 type wheat flour (ash, d.m. - 0.65%) and different proportions of defatted grape seeds flour were obtained, in the following ratios: 95:5, 90:10, and 85:15 (w/w). The types of flour mixtures used in this study are presented in table 1.

Table 1

THE TYPES OF MIXTURES OF FLOURS USED IN THIS STUDY

Category	Mixtures of flours	
	Wheat type 480	Grapes skin
P1	100%	-
P2	95%	5%
P3	90%	10%
P4	85%	15%
P5	-	100%

Chemical analysis

Moisture was determined for analytical DM by gravimetric loss of free water from heating to 103 ^{0}C (±2 ^{0}C) using test samples weighing 2 g, until constant weight was achieved between measurements, as it is described in the Official Mehod No. 110/1 [50]. The ash content was determined by incineration at 525 ± 25^{0}C, according to Official Mehod No 104/1) [50].

Total fat was determined by extracting 10 g of sample with petroleum ether 40-65 ^{0}C, using a semiautomatic Soxhlet Foss Extraction System 2055 (Foss, Sweden). Total nitrogen was analysed following Kjeldahl method (Official Method No. 950.36). Ash content (Official Method No. 930.22 [50]) in muffle furnace at 450-500^{0}C. Crude protein content was calculated by multiplying total nitrogen content by the factor 6.25. Crude fibers include cellulose, hemicellulose, and lignin. The crude fiber content of the samples was determined using a Fibretherm-Gerhardt apparatus.

The method for determination of crude fibers begins with treating the sample with an acid detergent solution (20 g N-cetyl-N,N,N-trimethylammonium bromide dissolved in 1 L H_2SO_4 0.5 M). In this solution, cellulose and lignin from the analysed material are insoluble, unlike all other components. Using special FibreBags, the dilution and filtration steps are simplified. The most important aspects of this method of analysis of the fibers are adherence to strict boiling times and to weighing procedures.

After treatment with the acid detergent solution, the insoluble residue is dried, weighed and then burnt. The acid detergent fiber (ADF) content represents the insoluble part of the sample that is left after boiling in acid detergent solution from which the ash obtained upon calcination is subtracted, and it is given by the equation:

$$\%ADF = \frac{[(\chi - \alpha) - (\delta - \xi)]100}{\beta}$$

Blank value (ξ)= $\gamma - \psi$

where: α - mass of FiberBag (g), β - sample mass (g), χ- mass of crucible and dried FibreBag, after digestion (g), δ - mass of crucible and and ash (g), ξ - blank value of empty FiberBag (g), γ - mass of crucible and ash of the empty FiberBag (g), ψ - mass of crucible (g).

Carbohydrate contents were calculated as the difference of 100-(ash+protein+fat+moisture). Each sample was analysed in triplicate.

Mineral content analysis

Mineral content was determined using an atomic absorption spectrophotometer (ContrAA 700; Analityk Jena). Total ash was determined by incineration at 550 ^{0}C, in an oven. Analysis was performed using an external standard (Merck, multi element standard solution) and calibration curves for all minerals were obtained using 6 different concentrations. Dried samples were digested in concentrated HCl.

Fatty acids profile

Using ^{1}H-NMR spectral technique, fatty acids composition was determined, especially the concentrations of short-chain saturated fatty acids (C_4-C_8), di-unsaturated fatty acids, mono-unsaturated fatty acids and long-chain saturated fatty acids (> C_8). ^{1}H-NMR spectra were recorded on a Bruker Ascend 400 MHz spectrometer, operating at 9.4 Tesla corresponding to the resonance frequency of 400.13 MHz for the ^{1}H nucleus. Samples were analyzed in 5 mm NMR tubes (Wilmad 507). The NMR samples were prepared by dissolving 0.2 mL oil in 0.8 mL $CDCl_3$. The chemical shifts are reported in ppm, using the TMS as internal standard.

Rheological properties evaluation

The rheological behavior of doughs was analyzed using the predefined *Chopin +* protocol on Mixolab, a new equipment of CHOPIN Technologies [51]. The international standard Standard Method No. 173 [50], a protocol for complete characterization of flours, was used, and a simplified graphic interpretation of the results was performed. The Mixolab is an apparatus used to characterize the rheological behaviour of dough subjected to a dual mixing and temperature constraint. It measures in real time the torque (expressed in Nm) produced by passage of the dough between the two kneading arms, thus allowing study of rheological and enzymatic parameters: dough rheological characteristics (development time, hydration capacity, etc.), protein reduction, enzymatic activity, gelatinisation and gelling of starch. The Mixolab can work with a constant dough weight to eliminate the influence of the mixer filling ratio [51].

The procedure parameters used for analysis of the rheological behavior in the Mixolab were as follows: tank temperature 30 ^{0}C, mixing speed 80 min^{-1}, heating rate 2 ^{0}C/min, total analysis time 45 min. Mixolab curves recorded (table 2) are essentially characterized by torque values in five defined points (C1-C5, N x m), temperatures and processing times corresponding to those points. The correlation between parameters (table 3) is tested during mixing and heating of dough by Mixolab.

The parameters obtained from the recorded curves are: water absorption (%) or percentage of water required for the dough to produce a torque (C1) of 1.1 N x m, mixing stability (min) or elapsed time at which the torque produced is kept at 1.1 N x m, protein weakening (C2, N x m and the difference between points C1-2, N x m), starch gelatinisation (C3, N x m and the difference between points C3-2, N x m), amylolytic activity (C4, N x m and the difference between points C3-4, N x m), starch gelling (C5, N x m and the difference between points C5-4, N x m).

Mixolab *Chopin +* transforms the standard curve into six quality indicators, expressed on a scale of 0-9 (Mixolab index) regarding:

- Water Absorption Index (a function of the composition of the flour (protein, starch, fiber). It affects dough yield. The higher the value, the more water is absorbed by flour.
- Mixing Index represents the behavior of the dough during mixing (stability, development time, and weakening). A high value corresponds to high dough stability in mixing.
- Gluten+Index represents the behavior of gluten when heating the dough. A high value corresponds to high gluten resistance to heating.
- Viscosity Index represents the increase in viscosity during heating. It depends on both amylase activity and starch quality. A high value corresponds to high dough viscosity during heating.
- Amylolysis Index, the starch's ability to withstand amylolysis. A high value corresponds to low amylase activity.
- Retrogradation Index represents the characteristics of starch and its hydrolysis during the test. A high value corresponds to a low shelf life of the end product.

Statistical analysis

All analyses were performed in triplicate and the mean values with the standard deviations were reported. Microsoft Excel 2003 Program was employed for statistical analysis of the data with the level of significance set at 95%. Analysis of variance (ANOVA) followed by Tukey's test was used to assess statistical differences between samples. Differences were considered significant for a value of $P < 0.05$.

Results and discussions

Chemical analysis of grapes skin flour, wheat flour and mixtures of these two

Grapes skin flour was chemically analysed to determine its contents of: proteins, ash, lipids, and crude fibers (Table 4). These data confirm that grapes skin flour is a good source of nutrients, especially crude fiber, which is the major

Point	Significance	Associated parameters* Temperature and time
C1	Used to calculate water absorption	T_1 and t_1
C2	Measures protein weakening as a function of mechanical work and temperature	T_2 and t_2
C3	Measures starch gelatinization	T_3 and t_3
C4	Measures the stability of the hot-formed gel	T_4 and t_4
C5	Measures starch retrogradation during the cooling period	T_5 and t_5

* *Dough Temperature (in ^{0}C) and the time (in min) taken for different types of torque to appear*

Table 2
MIXOLAB CURVES INTERPRETATION

Parameter	Calculation method	Significance
Water absorption %	Quantity of water required to obtain C1=1.1 Nm +/- 0.05	Quantity of water that the flour can absorb to achieve a given consistency during the constant temperature phase
Time for C1 (min)	Time required to obtain C1	Dough formation time: The stronger the flour, the longer it takes.
Stability (min)	Time during which torque is > C1 – 11% (constant T0 phase)	Dough resistance to kneading: The longer it takes the "stronger" the dough.
Amplitude (Nm)	Curve width at C1	Dough elasticity: The higher the value, the greater the flour elasticity.

Table 3
MIXOLAB PARAMETERS CORRELATION AND SIGNIFICANCE

Composition % d.m.	Samples				
	P1	P2	P3	P4	P5
Protein	13.2 ± 0.24	13.3 ± 0.25	13.40 ± 0.25	13.50 ± 0.21	15.26 ± 0.20
Ash	0.49 ± 0.01	0.85 ± 0.02	1.25 ± 0.02	1.63 ± 0.03	7.95 ± 0.04
Total Fat	1.04 ± 0.07	1.25 ± 0.06	1.49 ± 0.07	1.71±0.07	5.44 ± 0.08
Carbohydrate	85.27 ± 0.12	84.57 ± 0.12	83.88 ± 0.11	83.18±0.11	71.35 ± 0.11
Crude Fiber	1.9 ± 0.12	2.49 ± 0.35	3.05 ± 0.38	3.63 ± 0.56	13.28 ± 0.78

Table 4
COMPONENTS OF WHEAT FLOUR, GRAPE SKIN FLOUR, AND THEIR MIXTURES

component (15.26% d.m.). Grapes skin should be regarded as an interesting source for enriching bread and other bakery products in carbohydrates, particularly crude fibers with known prebiotic properties, useful in the formulation of functional foods, as well as nutraceuticals [3].

The compositions of wheat flour, grapes seed flour, and mixtures of the two are shown in table 4. The ratios of the different flours that were incorporated were shown in Table 1. It is apparent that the enrichment of wheat flour with nutritionally rich grape seeds flour enhances the nutritional qualities of bakery products.

It can be observed that P3 sample (95% wheat flour+10% grape skin flour) contains more than 3 grams of crude fiber per 100 g total, which allows the provision of nutritional term *source of fiber*. These data (fig. 1) confirm that grape skin flour is a good source of bio-compounds, especially crude fibers (13.28%, d.m). Grape skin should be considered a source of interest for adding value to carbohydrate compounds with known potential prebiotic properties, useful to formulate functional foods as well as nutraceuticals.

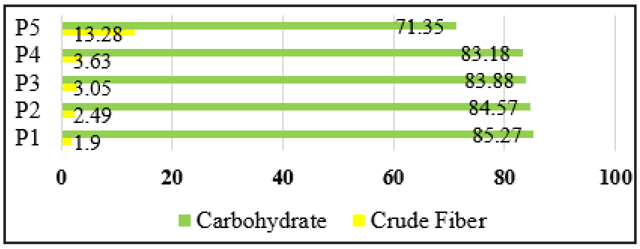

Fig. 1. Crude fiber and carbohydrate content (in %) in mixes of flours

Effect of incorporation of grape skin flour on the minerals content of wheat flour

In the present study, the contents of four biologically essential mineral elements were analyzed: calcium, magnesium, potassium, iron, and one essential trace elements, copper.

The mineral contents of the samples are given in table 5. Their profiles in the samples of wheat flour and grape skin flour were typical for these plant species.

From performed analyses regarding minerals content it can be observed that grape skin flour is a material having important minerals content. It is easily noticeable that, compared to the low mineral content of the wheat flour sample (P1), mixtures of wheat flour and grape skin flour have higher contents of minerals, in direct proportionality with the percentage increase of grape skin flour added in the flour mixtures.

Daily dose of copper (RDI) recommended by the FDA (2011) is 2 mg. It is easily noticeable that the wheat flour mixtures with 10 and 15%grape skin flour fulfill this recommendation, therefore, these flours can be regarded as a valuable *Source of copper* (FDA 2011).

The fatty acids profile of samples properties of flour mixtures

In table 6 is presented the fatty acids profile of samples. The addition of partially defatted hemp seed in flour mixtures modifies the lipid content of samples compared to the control sample P1.

The high nutritional value of grape skin flour, (total fat – 13.28% d.m.), their complex physiological effect and the wide range of possible uses can be attributed to their substantial oil contents and to their favorable fatty acid compositions. The addition of grape skin flour in flour mixtures modifies the total unsaturated fatty acids content compared to the control sample P1 (fig.2).

Taking into account that consumers are more and more aware about the food quality, especially from the nutritive point of view, the new food resources rich in bioactive compounds are necessary to be found. In this respect,

Table 5
MINERAL CONTENT OF WHEAT FLOUR, GRAPE SKIN FLOUR AND MIXTURES THEREOF

Sample	Mineral content (mg mineral/100 g sample)				
	Ca	Mg	K	Cu	Fe
P1	43.8 ± 0.59	47.7 ± 0.55	187 ± 0.75	0.78 ± 0.09	1.1 ± 0.22
P2	66.5 ± 0.63	50.0 ± 0.55	301.8 ± 0.87	1.38 ± 0.10	1.67 ± 0.25
P3	89.1 ± 0.79	52.2± 0.57	416.1 ± 0.99	1.97 ± 0.19	2.21 ± 0.28
P4	111.3 ± 0.89	54.5 ± 0.59	531.2 ± 1.01	2.54 ± 0.22	2.76 ± 0.30
P5	476 ± 1.29	94 ± 1.01	2480 ± 2.65	12,5 ± 1.14	12.1 ± 0.75

Table 6
FATTY ACIDS PROFILE USING NMR SPECTROSCOPY (G/100 LIPIDES)

Sample	Fatty acids %				
	Short-chain saturated	Mono-unsaturated	Di-unsaturated	Long-chain	Total unsaturated
P1	20.75	24.76	44.85	9.64	79.25
P2	20.66	25.19	44.99	9.16	79.34
P3	20.58	25.63	45.12	8.68	79.42
P4	20.49	26.06	45.26	8.19	79.51
P5	19.02	33.43	47.55	0	80.98

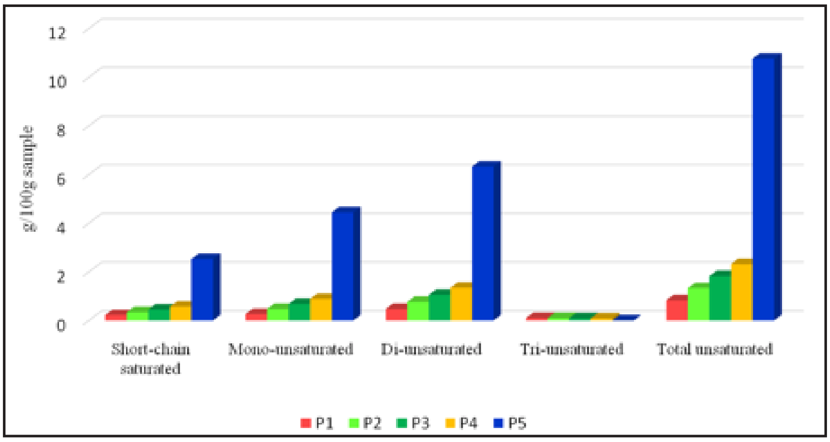

Fig. 2. The influence of the addition of grape skin on the fatty acid content of the mixtures of flour

grape skin meets the expectations of such consumers. It is observed that with increasing addition of the grape skin flour increases the content of total unsaturated fatty acids (P4).

Rheological properties of flour mixtures

The rheological behavior of wheat flour dough (P1) and of all flour mixtures during the Mixolab test is illustrated in Table 7. Mixolab C1-C5 values of pure wheat dough (P1) were 1.10 N x m, 0.454 N x m, 2.327 N x m, 2.216 N x m, and 3.664 N x m, respectively.

Following the results, it can be seen that, as the amount of added grape skin flour increases, the water absorption capacity (CH) increases from 56% (P1) to 56.5% (P2), 57.2% (P3) and 57.8% (P4), respectively. As such, with regard to water absorption capacity, the baking characteristics of flours did not decrease considerably as the grape skin flour content increased. For bread flour, optimal CH values are between 55%-62% [52].

Dough stability had the following values: 10.07 min (P1), 10.28 min (P2), 10.73 min (P3) and 12.23 min (P4). It can be noticed that the addition of grape skin flour did not have a significant influence on the stability of the wheat flour sample, even for the sample with 15% added grape skin flour.

Addition of grape skin flour did not significantly influence the amplitude, i.e. the width of the curve during dough formation, this being higher for the 15% grape skin flour (P4), which suggests a higher elasticity of the dough, due to higher content of fat. This increase in fat content has a positive influence on doughs. As the percentage of grape skin flour increased, the amplitude, i.e. the width of the curve during dough formation, increased, which suggests a higher elasticity of the dough, due to higher content of fat. This increase in fat content has a positive influence on doughs.

As the content of grape skin flour increases, dough formation time (TC1) also increases (table 7). A bigger resistance of dough to mixing is noticed.

A small increase of consistency C2 (the degree of dough weakening as a consequence of temperature) means some small negative qualitative changes in flour protein composition, i.e. dilution of gluten content and changes of gluten structures, can possibly occur.

In phase 3, the starch gel formation, when the temperature reached 50-55 ^{0}C, the biggest C3 was observed for P4. The difference in C3 results between P1 and P4 samples was 0.16 N x m, so the influence of dough preparation recipe was low. The difference in C3 for consecutive samples is rather small, such that the influence of percentage of skin flour for the two mixtures (P2 and P4) on the rheological quality of flour is minor. This will be tested by production of bakery products.

As mentioned above (table 2), the C4 parameter corresponds to the stability of the starch gel formed. In this sense, a dependence of determined values on dough formulation was sought. The lowest C4 was found for P4 (table 7). The difference of C4 results between P1 and P4 samples was only 0.059 N x m, thus insignificant. The stability time of the gel (TC4) decreases as the percentage of grape skin flour increases.

The retrogradation stage of starch (C5) for the tested wheat flour and wheat-grape skin flour mixtures demonstrated similar differences as for starch gel stability. It can be seen that differences in C5 between consecutive samples are generally not significant, but some difference between P1 and P4 is registered (3.664 and 3.008 N x m, respectively).

From all of the above data, it can be stated that, with regard to their baking characteristics, these flour mixtures fall into the category of flours suitable for bakery products. In figure 3 is presented the influence of substitution level of grapes skin flour on the Mixolab curve.

Table 7
INFLUENCE OF GRAPE SKIN FLOUR ADDED TO WHEAT FLOUR IN DIFFERENT PROPORTIONS ON MIXOLAB CHARACTERISTICS (RHEOLOGICAL BEHAVIOR)

Parameter	Abrev. name	P1	P2	P3	P4
Water absorption (%)	CH	56.0	56.5	57.2	57.8
Stability (min)	ST	10.07	10.28	10.73	12.23
Amplitude (Nm)	A	0.103	0.093	0.095	0.117
Maximum consistency during:					
– phase 1 (N·m)	C1	1.10	1,111	1,099	1,097
	TC1	5.55	6.37	8.68	10.45
– phase 2 (N·m)	C2	0.454	0.469	0.486	0.667
	TC2	16.95	16.68	17.28	17.72
– phase 3 (N·m)	C3	2.327	2.432	2.491	2.490
	TC3	24.53	24.43	24.08	23.17
– phase 4 (N·m)	C4	2.216	2.258	2.296	2.275
	TC4	27.58	26.78	26.37	26.01
– phase 5 (N·m)	C5	3.664	3.607	3.221	3.008
	TC5	45.02	45.02	45.2	45.02

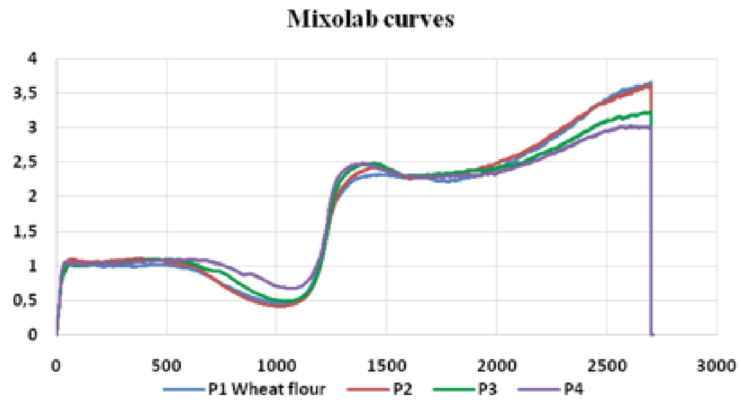

Fig. 3. The influence of substitution level of grapes skin flour on the Mixolab curve

Conclusions

The compositional characterization performed in this study proved that the grape skin flour is a valuable source of crude fiber, fat with a high percentage of unsaturated fatty acids and minerals, especially calcium, magnesium, iron potassium and copper.

The main conclusion in our study concerning the rheological properties of dough (pure wheat flour and mixtures of wheat flour with grape skin flour) is that rheological parameters were maintained within 15% limits that can assure a good technological behavior towards obtaining high quality bakery products.

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