

Oscillatory Rheology and Creep-Recovery Behaviour of Grape Seed-Wheat Flour Dough: Effect of Grape Seed Particle Size, Variety and Addition Level

Mădălina IUGA¹, Costel MIRONEASA², Silvia MIRONEASA^{1*}

¹Faculty of Food Engineering, Stefan cel Mare University of Suceava, 13th University Street, 720229, Suceava, Romania

²Faculty of Mechanical Engineering, Mechatronic and Management, Stefan cel Mare University of Suceava

*corresponding author: silviam@fia.usv.ro; silvia_2007_miro@yahoo.com

Bulletin UASVM Food Science and Technology 76(1)/2019

ISSN-L 2344-2344; Print ISSN 2344-2344; Electronic ISSN 2344-5300

DOI: 10.15835/buasvmcn-fst: 2018.0020

Abstract

In the present study, the influence of grape seeds flour (GSF) addition on the physico-chemical and white wheat flour dough rheological properties were investigated. GSF of two grape varieties, white and red, was added at three particle sizes (L, M and S) and different addition levels (0, 3, 5, 7, 9%) to the white wheat flour. The fundamental rheological properties were investigated by applying oscillatory tests using a dynamic rheometer. The storage (G') and loss (G'') moduli in function of frequency and temperature respectively, were measured. Creep-recovery tests were also applied to evaluate grape peels-wheat flour dough behaviour. The results showed that the GSF incorporation increased protein content and alpha-amylase activity in composite flour with particle size decrease, affecting gelatinization temperature. The changes in dough viscoelasticity depend on the particle size, addition level and grape variety, factors which affected dough capacity to deform. The effects of GSF from white grape were more pronounced compared to GSF from red grape variety. GSF addition exhibited an increase in values of both dynamic moduli, which was more pronounced for the white variety than for the red one. The knowledge of this information is useful in new products development, especially for bakery products manufactory.

Keywords: grape seeds, particle size, rheology, wheat flour

Introduction

In the development of innovative products, new sources of ingredients such as vegetables or fruit-processing by-products have been investigated for their potential to increase the nutritional value of cereal products. Bread is a convenient food based on wheat consumed worldwide (Bakke and Vickers, 2007), generally made from white flour which has low dietary fiber content, can serve as a matrix for new functional food development. Incorporation of functional compounds such as dietary fibre and bioactive compounds in refined wheat flour had a great attention in recent years to fulfil the increasing demand for eating healthy and maintaining good health at affordable price.

Grape seeds, a solid by-product generated by wine industry, are being used increasingly to obtain ingredients, such as extract, oil and flour which have the potential to provide a wide range of food products with numerous health benefits (Lavelli et al., 2016). They represent between 2 and 5% of grape weight (Brenes et al., 2016) and contains about 40% fiber, 10-20% lipid, 10% protein, complex phenolics, sugars and minerals (Mironeasa, 2017; Rockenbach et al., 2012). Grape seeds are recognized for their nutritional properties of the oil, which is characterized by higher levels of unsaturated fatty acids and phenolics compounds (Choi et al., 2010; Bail et al., 2008). In the fatty acid profile polyunsaturated fatty acids such as linoleic

and linolenic acids are predominant (58%–78%), being essential for the human metabolism, whereas the proportion of monounsaturated and saturated fatty acids are 14.2%–21.3% and 11.6%–14.9%, respectively (Fernandes et al., 2013). Grape seed oil has a high vitamin E content (85.5 to 260.5 mg/kg oil) (Fernandes et al., 2013) which helps to reduce the risk of suffering from arteriosclerosis and to decrease cholesterol values, its intake may be beneficial to prevent heart and circulatory problems (Bail et al., 2008). Grape seeds are also valued for the phenolics compounds, such as anthocyanin, catechin, and gallic acid, the most important grape secondary metabolites with antioxidant properties. The phenolic content may range from 5% to 8% by weight of seeds (Baiano and Terracone, 2011). The consumption of polyphenol-rich food has been associated with diseases prevention, such as different types of cancer and coronary heart conditions (Scalbert et al., 2005). Several works reported in the literature the bioactive compounds applications in food, pharmaceutical, cosmetics and others products and their health effects (García-Lomillo et al., 2014; Guerra-Rivas et al., 2016). Grape seed is a potential source of dietary fibers (DF) with a high insoluble DF content (Mironeasa, 2017; Iuga et al., 2017; Mironeasa et al., 2017), better insoluble/soluble fiber ratio and low caloric content. Grape seeds DF are so-called “antioxidant dietary fibre”, defined by Saura-Calixto (1998) as a product that is composed of more than 50% of dietary fiber on a dried matter basis and present a natural antioxidant capacity equivalent to at least 50 mg of vitamin E. Antioxidant dietary fiber is of great interest for various food applications due to its benefits for human health. Grape seed contain also mineral substances such as calcium, potassium phosphorous, magnesium, iron, manganese and zinc, copper (Amariei et al., 2018; Mironeasa et al., 2010) very important nutrients for the human body.

In recent years, grape seeds in the form of flour or powder, was used in food industry as an ingredient in different products such as cereal bars (Balestro et al., 2011), biscuits and cookies (Mildner-Szkudlarz et al., 2013; Piovesana et al., 2013; Acun and Gül, 2014), extruded snacks (Bender et al., 2016.), and muffin (Bender et al., 2015) to develop products rich in fibre, with antioxidant capacity potential and good acceptances by consumers. As gluten-free ingredient, grape seeds flour can be used as a

biologically active substance in food consumed by people who suffer from celiac disease.

Dough rheological characteristics have an essential effect on bread baking properties and, as well are determinants for the design of equipment and processes. Most researchers used empirical dough testing device such as Farinograph, Amilograph, Alveograph to study the rheological properties of wheat flour dough. Recently, the dynamic test and creep-recovery test have been used in various studies to evaluate the fundamental mechanical properties of wheat flour doughs which are more significant for dough handling behaviour during processing understanding or for clarifying the interactions among dough components (Berland and Launay, 1995).

The use of grape seeds flour as dietary fiber source in white wheat flour in order to develop the new bread-making products modifies dough rheological characteristics, enhancing the quality of the product, as function of particle size. The effect of GSF in refined wheat flour dough with different particle size must be tested to improve the rheological characteristics. Particle size has an important role on the rheological behaviour of dough, influencing viscosity and bakery product quality (Ahmed et al., 2016). The knowledge of particle size influence on the functional and structural properties is needed to confirm the suitability of their use in bread products development and process engineering. To the authors knowledge there are some studies on the influence of grape seeds on the dough rheological parameters (Mironeasa et al., 2012; Mironeasa et al., 2014; Aghamirzaei et al., 2015; Mironeasa et al., 2017; Iuga et al., 2017) but are not dealing with two varieties of grape seeds, at three different particle sizes additions in wheat flour and the addition levels did not have the same concentrations.

Thus, the purpose of this study was to investigate the effects of three different GSF particle sizes, large ($L > 500 \mu\text{m}$), medium ($200 \mu\text{m} > M < 500 \mu\text{m}$) and small fractions ($S < 200 \mu\text{m}$) from two grape variety (white and red), additions at levels of 0, 3, 5, 7 and 9% in refined wheat flour on the physicochemical composition and dynamic rheological properties of dough. Dynamic rheological analysis was performed by applying the oscillatory and the creep-recovery tests using a dynamic Haake MARS Rheometer. To our knowledge, no report has been evaluated

and compared the viscoelastic properties of wheat dough supplemented with three different particle sizes of grape seed from two varieties at five levels.

Materials and methods

Basic materials

Refined wheat flour of 550 flour type (harvest 2016) with 13.80% moisture content (ICC methods 110/1), 0.55% ash content (ICC 104/1), 11.05% protein content (ICC 105/2), 28.0% wet gluten content (ICC 106/1), 4.00 mm gluten deformation index (SR 90:2007) and a falling number index of 354.5 s (ICC 107/1) was supplied by S.C. Dizing S.R.L. (Brusturi, Neamt, Romania). White wine grape pomace and red wine grape pomace were provided by the viticulture center Jaristea, Odobesti ecosystem. Grape seeds were separated manually from dried grape pomace in open air, ground in a domestic blender and sieved through a Retsch Vibratory Sieve Shaker AS 200 basic (Haan, Germany) to obtain grape seeds flour (GSF) at three different particle sizes: large, $L > 500 \mu\text{m}$, medium, $200 \mu\text{m} > M < 500 \mu\text{m}$ and small fractions, $S < 200 \mu\text{m}$. The characteristics of seeds, from white and red grape variety were: 7.70% and 7.60% moisture content (SR EN ISO 665:2003), 18.32% and 20.75% fat content (SR EN ISO 659:2009), 9.79% and 12.17% protein content (SR EN ISO 20483:2014), 2.79% and 2.59% ash content (SR ISO 2171:2010). Fiber content, 40.80% and 45.20% was determined by Near Infrared Reflectance Spectroscopy (NIR) with a FOSS machine (model 6500, Denmark) using the AUNIR calibration.

The GSF fractions (L, M and S) were incorporated in wheat flour at different substitution levels (3, 5, 7 and 9%), achieving the following coded samples: 3L, 3M, 3S, 5L, 5M, 5S, 7L, 7M, 7S, 9L, 9M and 9S. The sample with 0% GSF was used as control. The dough samples used for further analyses were prepared an optimal dough water absorption established by Farinograph.

Fundamental dough rheological properties

The fundamental rheology measurements were done by the use of a Haake dynamic rheometer (MARS 40, Termo-Haake, Karlsruhe, Germany). It was chosen a plate-plate geometry, at a constant temperature of $20.0 \pm 0.1^\circ\text{C}$, with a gap of 2 mm. The dough sample was fixed between the plates, the excess dough was carefully removed, and a

thin layer of vaseline was applied to the exposed sample surface to prevent loss moisture during testing. Samples were let to rest between plates during 5 min before conducting the measurements. The measurements were conducted in the linear viscoelastic region (LVR). In this sense, first step of the measurements was strain sweep tests (0.01%-10%) at constant oscillation frequency of 1 Hz, without any damage of the dough, in order to establish LVR. Next, frequency sweep test from 1 to 20 Hz at 15 Pa stress, in the LVR, for the samples formulated was applied to determine the storage modulus (G') and the loss modulus (G''). The temperature sweep tests were then performed at a constant strain of 0.15% and a frequency of 1 Hz, dough being heated from 20 to 100°C at a heating rate of $4.0 \pm 0.1^\circ\text{C}$ per min. The storage modulus (G') and loss modulus (G'') were recorded as a function of temperature. To simulate different stress during bread dough production, the oscillation test with small forces was complete with a creep-recovery test at force which is still in the LVR, the dough being kept under a pressure of 50 Pa for 60 s and after removing the pressure there was a relaxation time of 180 s. The maximum compliance ($J_{c_{\max}}$) value reached in the creep phase for 60 s, which corresponds to the maximum deformation and the maximum compliance value at the end of the recovery phase ($J_{r_{\max}}$), related to partial reformation after stress removal, were reported. The results of creep-recovery measurements are expressed as compliance, which corresponds to the strain divided by the imposed shear stress of 50 Pa. Also, the ratio between the compliance value at the end of the recovery phase (180 s) and the maximum creep compliance value reached in the creep phase for the 60 s was determined, as recovery percent, $\text{Recovery (\%)} = J_{r_{\max}}/J_{c_{\max}}$ (Abebe et al., 2015). The experiments were conducted in at least two replications for each sample formulated.

The statistical analysis of the experimental data was done and results were expressed as mean value with the standard deviation. The analysis of variance (ANOVA) and the *post-hoc* multiple comparisons with subsequent least significant difference (LSD) test at a significance level of $p < 0.05$ (SPSS version 16.0) in order to evaluate the difference between the parameters of the different formulations was performed.

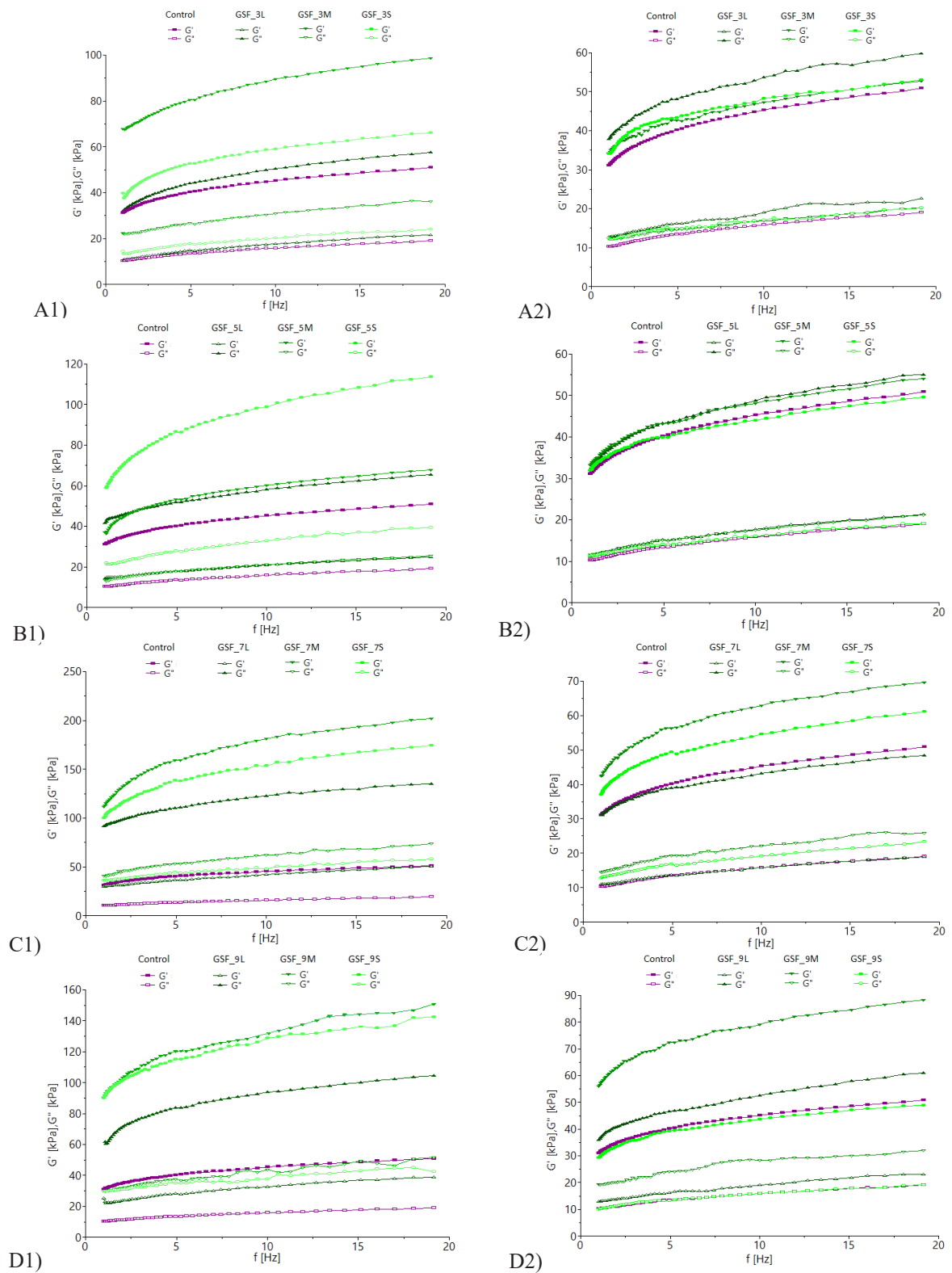


Figure 1. Effect of grape seed flour (GSF) addition from white (A1-D1) and red (A2-D2) grape varieties at different levels (3, 5, 7 and 9%) and particle size (L, M and S) as compared to control sample (0% GSF) on changes of the storage modulus (G') and loss modulus (G'') vs. oscillation frequency (f)

Results and discussion

The effect of grape seeds on the chemical composition of wheat flour

GSF addition in white wheat flour leads to changes in the chemical composition and falling number index values. Table 1 summarizes mean and standard deviation values for physico-chemical properties of samples formulated in function of particle size, addition level and grape seed variety.

For both GSF additions in wheat flour, it may be seen an increase in ash and protein content with GSF particle size decrease, the higher values were obtained for S particle size. This increase may be due to the ash and protein content of S particle size of GSF which is different as compared to the M and L particle sizes. Statistically significant difference was obtained for protein content between samples with L M and S particle sizes ($p < 0.05$), for both grape seeds varieties. Our

results showed that the samples with GSF from each variety had a significant increasing effect ($p < 0.05$) of ash and protein contents with the increase of addition levels. The ash and protein content were significantly higher ($p < 0.05$) in samples with GSF from red grape variety comparing to the GSF from white variety (Table 1). The moisture content increased with decreased of GSF particle size from white variety at addition levels above 5%, being lower comparing to the control sample. On the other hand, a decrease of moisture with the decrease of GSF particle size from red variety addition was found which can probably be due to the moisture difference between the white grape seeds flour and the red one. In respect to the addition level, the moisture content significantly ($p < 0.05$) decreased with the increase of GSF level addition from both grape seeds varieties.

The falling number index (FN) values decreased with the decrease of GSF particle size addition for both types of grape varieties,

Table 1. The physico-chemical properties of white wheat flour as influenced by grape seed flour addition from two grape varieties at different levels and particle size

Sample	GSF from		GSF from		GSF from		GSF from	
	white variety	red variety	white variety	red variety	white variety	red variety	white variety	red variety
	Moisture (%)		Ash (%)		Protein (%)		Falling number (s)	
Control	13.75±0.05 ^A	13.75±0.05 ^A	0.58±0.00 ^A	0.58±0.00 ^A	11.05±0.07 ^A	11.05±0.07 ^A	354.50±10.00 ^A	354.50±10.00 ^A
GSF_3L	13.00±0.05 ^{abx}	13.15±0.07 ^{bbx}	0.72±0.00 ^{abx}	0.76±0.02 ^{bbx}	11.10±0.00 ^{abx}	11.20±0.00 ^{abx}	353.00±0.00 ^{ax}	352.00±4.00 ^{ax}
GSF_3M	12.88±0.03 ^{aby}	13.10±0.00 ^{bbx}	0.79±0.00 ^{aby}	0.88±0.02 ^{by}	11.20±0.00 ^{aby}	11.35±0.06 ^{by}	348.00±2.00 ^{ay}	351.00±1.50 ^{ax}
GSF_3S	12.75±0.07 ^{abz}	12.90±0.00 ^{by}	0.88±0.00 ^{abz}	0.99±0.00 ^{bz}	11.35±0.07 ^{abz}	11.70±0.00 ^{bz}	344.00±5.50 ^{ay}	342.00±4.50 ^{ax}
GSF_5L	12.60±0.00 ^{acx}	13.30±0.30 ^{acx}	0.73±0.01 ^{acx}	0.85±0.00 ^{bcx}	11.10±0.00 ^{acx}	11.25±0.07 ^{bcx}	352.50±7.50 ^{bx}	334.00±5.50 ^{bx}
GSF_5M	12.68±0.03 ^{acy}	13.10±0.50 ^{acx}	0.89±0.02 ^{acy}	1.06±0.02 ^{bcy}	11.35±0.07 ^{acy}	11.60±0.14 ^{bcy}	342.50±10.00 ^{by}	329.00±4.50 ^{bx}
GSF_5S	12.90±0.05 ^{acz}	12.80±0.20 ^{acy}	1.02±0.00 ^{acz}	1.20±0.03 ^{bcz}	11.50±0.14 ^{acz}	11.90±0.00 ^{bcz}	342.00±3.00 ^{by}	331.50±12.00 ^{bx}
GSF_7L	12.60±0.00 ^{adx}	13.20±0.15 ^{bdx}	0.74±0.01 ^{adx}	0.93±0.02 ^{bdx}	11.20±0.00 ^{adx}	11.50±0.00 ^{adx}	352.50±3.50 ^{cx}	327.50±5.00 ^{bx}
GSF_7M	12.68±0.00 ^{ady}	13.05±0.05 ^{bdx}	1.04±0.00 ^{ady}	1.26±0.00 ^{bdy}	11.45±0.07 ^{ady}	11.90±0.15 ^{bdy}	341.50±5.00 ^{cy}	326.50±5.00 ^{bx}
GSF_7S	12.80±0.05 ^{adz}	12.75±0.10 ^{ady}	1.17±0.04 ^{adz}	1.38±0.02 ^{bdz}	11.70±0.15 ^{adz}	12.10±0.00 ^{bdz}	339.00±4.50 ^{cy}	324.00±8.50 ^{bx}
GSF_9L	12.05±0.07 ^{hex}	12.85±0.07 ^{hex}	1.10±0.02 ^{hex}	0.94±0.04 ^{hex}	11.45±0.07 ^{hex}	11.58±0.04 ^{hex}	351.50±9.00 ^{dx}	320.50±2.50 ^{bx}
GSF_9M	12.55±0.07 ^{hey}	12.80±0.10 ^{hex}	1.13±0.01 ^{hey}	1.40±0.02 ^{hey}	11.65±0.07 ^{hey}	11.95±0.08 ^{hey}	340.50±5.00 ^{dy}	318.00±8.50 ^{bx}
GSF_9S	12.60±0.15 ^{hez}	12.75±0.10 ^{hey}	1.37±0.02 ^{hez}	1.42±0.03 ^{hez}	11.95±0.07 ^{hez}	12.15±0.06 ^{hez}	338.00±5.50 ^{dy}	314.00±11.00 ^{bx}

Grape seeds flour (GSF) particle size: Large, L > 500 μ m, Medium, 200 μ m > M < 500 μ m and Small fractions, S < 200 μ m. Mean values of triplicates (\pm standard deviation) for independent evaluated samples. Mean values are followed by different superscript (a-b) in the same row (GSF from white/red variety) if are significantly different, according to Turkey's test ($p \leq 0.05$); A-E for the different addition level (3, 5, 7 and 9%) of GSF, means in the same column with different superscript were significantly ($p \leq 0.05$); x-z for the same addition level of GSF and different particle sizes (L, M and S), means in the same column with different superscript were significantly ($p \leq 0.05$).

indicating an increase of alpha-amylase activity with particle size decrease in the flour blends. This effect is due to the fact that alpha-amylase is a metalloenzyme which depends on the presence of metal ions calcium in its molecule for its activity (Sundarram et al., 2014). It is known that within macro elements, calcium is the most abundant in the grape seeds (Ahn and Son, 2012; Mironeasa et al., 2010) and therefore, an increased level of GSF in GSF-wheat flour mix will lead to an increase of alpha-amylase activity. This increase of alpha-amylase activity, especially in the flour with low alpha-amylase activity, conducts to an improved quality of the final bakery products due to the fact that alpha-amylase provides fermentable sugars for the yeast, increasing bread volume, crust color, improving crumb structure, e.g. A similar trend for the FN values variation was also reported by Mironeasa et al., (2012; 2014). Significant differences ($p < 0.05$) was obtained for FN index between samples with L and M, S particle sizes from white grape variety, while the differences between particle sizes from red grape variety was non-significant ($p > 0.05$). Compared to the control sample, the FN index significantly varied ($p < 0.05$) in samples with 5, 7 and 9% GSF addition level for both grape variety (Table 1).

Fundamental dough rheological properties

The dynamic rheological properties determined for white wheat flour dough with different particle sizes and different levels of GSF from white and red grape varieties are shown in Figure 1 (A1-D1) and (A2-D2) for frequency sweep tests, while the evolution of dynamic modulus with temperature is shown in Figure 2 (A1-D1) and (A2-D2).

The experimental data of storage modulus (G'), as a measure of the solid or elastic character of the dough, and loss modulus (G'') as a measure of the liquid or viscous character, for all dough samples formulated with GSF at different particle sizes and addition levels, as it was expected, showed $G' > G''$. An increase of dynamic moduli values with the increase of oscillation frequency within the range from 1 to 10 Hz can be observed for all samples (Figure 1). This behaviour can be attributed to the presence of binding agents in the mix flour doughs and attractive forces between starch granules are predominant. The loss modulus exhibit lower values than the storage modulus in the whole range of frequency, suggesting a solid, elastic-like

behaviour of all dough samples under the testing conditions. Both moduli have higher values for white GSF mixes as compared to the red ones. G' and G'' values of composite flour dough were higher, in the case of grape seed from white variety, than those reported for wheat flour (Salvador et al., 2006). The increase of moduli might be attributed to different interactions between starch granules with different particle sizes of GSF. The addition of GSF enhanced elasticity of the dough differently. The particle sizes of the different addition levels do not have a similar trend. For example, for the S particle size, the 7 and 9% addition levels cause a higher increase of G' compared to 3 and 5% additions for white GSF.

The results indicate that the additions of GSF at different levels and particle sizes caused an increase in bread dough elasticity (G') and viscosity (G''), especially for dough samples with white GSF, G' was greater than G'' , in the whole range of frequencies. Both moduli slightly increased with frequency for all dough formulations, in dependency on the GSF concentration and particle size, suggesting a solid elastic-like behaviour of the GSF-wheat flour dough formulations. The elasticity increase dominates over that of viscosity, obtaining significantly higher values for composite flour dough than for wheat flour dough, according to the results reported by Lamacchia et al., (2010) for oat wholemeal dough and wheat dough.

All the particle sizes applied at a level of 3%, for both varieties, led to a similar trend of the moduli. Higher G' values were obtained for 3M addition of white GSF than of red GSF. As it is evident in Figure 1B2, the addition of GSF from red variety caused a lowering of dough elasticity as it can be seen from the drop of G' for the GSF_5S-wheat flour mix as compared to the control sample, while the dough viscosity (G'') has a value closed to the control wheat flour dough, in according to the results obtained by Moreira, (2010) for smallest particle size of chestnut flour. Dough elasticity increases for S particle size at 7% addition level (GSF_7S) (Figure 1C2), but at 9% it decreases again close to the control (Figure 1D2). This fluctuations can be explained by the differences between the hydration capacities of the flour mixes. The decrease is more prominent at higher frequency. Also, the different interactions between starch granules and GSF particle size have an influence, the structure and

chemical composition of the particle size can be related to this variations.

The different temperature sweep curves for the samples with GSF at different levels

and particle sizes added in white wheat flour are shown in Figure 2. As it can be observed, all dough samples with GSF showed similar thermomechanical behaviour as wheat flour

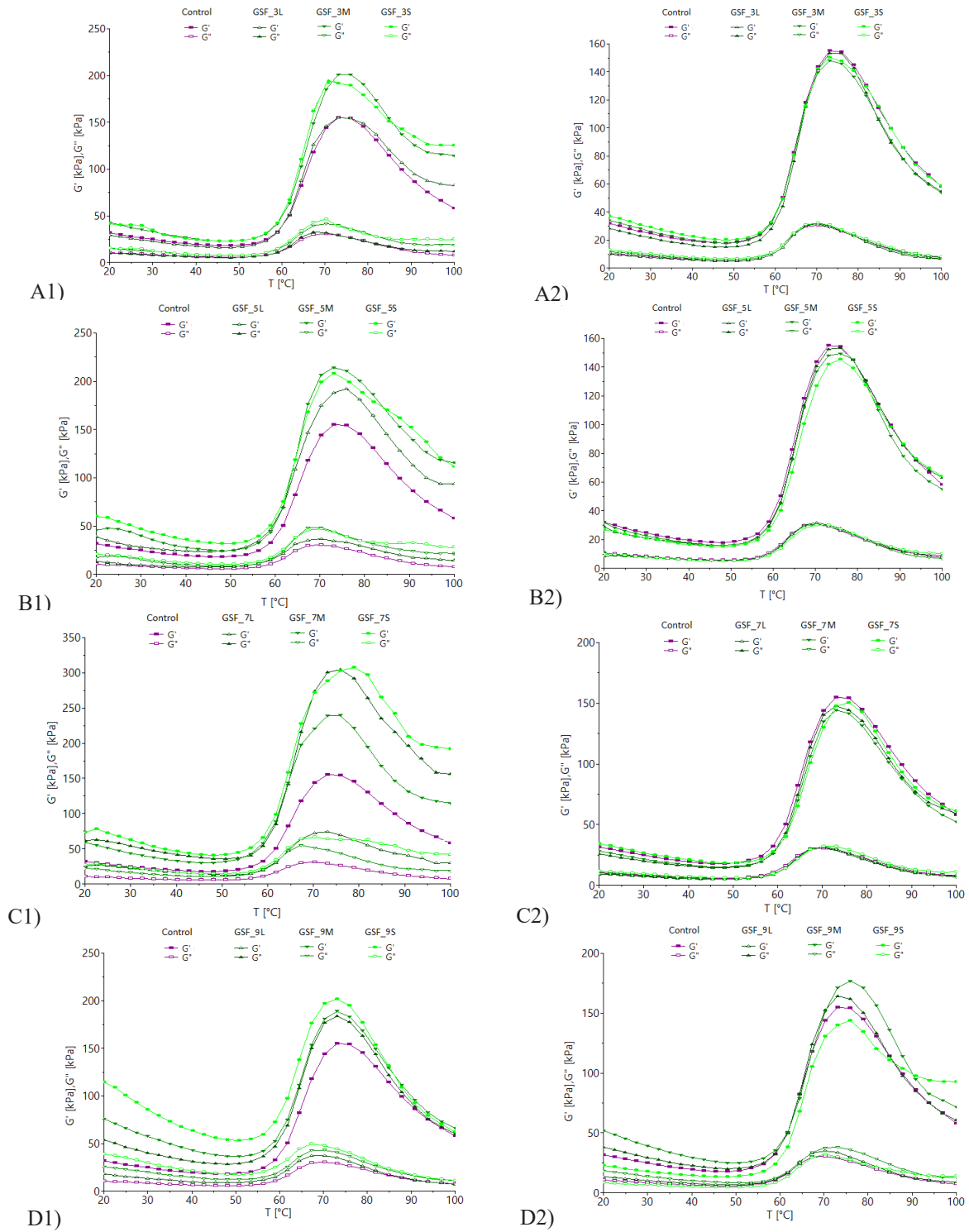


Figure 2. Effect of grape seed flour (GSF) addition from white (A1-D1) and red (A2-D2) grape varieties at different levels (3, 5, 7 and 9%) and particle size (L, M and S) as compared to control sample (0% GSF) on changes of the storage modulus (G') and loss modulus (G'') vs. temperature (T)

dough control. The changes in dynamic moduli, G' and G'' , as the temperature increases highlights three distinct stages for dough samples. The first stage indicates a decrease of the moduli values at low temperature due to the proteins denaturation which lose their capacity to retain water and are linked gradually by the starch granules which gelatinize, leading to the rises of dough viscoelastic properties. This rise is related to the content of alpha-amylase from wheat flour and mix flours, respectively, a higher content of amylase will cause a lower increase (Codină et al., 2012). In the second stage, G' increases rapidly until achieves a maximum value due to the swell rising of starch granules indicating the gelatinization of starch. Dough viscosity and elasticity increased, amylose leached out into the aqueous intergranular phase during the gelatinization process (Addo et al., 2001). Starch gelatinization was finished in the third stage and the dynamic moduli began to decrease due to starch degradation. After adding GSF, the temperatures at which the maximum value of G' and G'' was observed were dependent on the particle size and level of GSF. An increase of modulus with the addition level increase of GSF from white grape variety was observed for samples with S particle size. This increased can be related to the protein content higher in S so than to the M and L particle sizes. In agreement with Eliasson and Gudmundsson, (1996), protein content has a greater influence on the changes induced by heat during the first heating stages, until the inflection point is reached, after which the effects of the gelatinised starch become dominant. The presence of protein remarkable affected rheological changes in the dough during heating, increasing gluten cross-linking, during complete starch gelatinization the G' increases rapidly, obstructing other effects (Weipert, 1990). Compared to the control sample, the peak value of G' and G'' significantly increased in case of added GSF from white grape variety and slightly decreased for red grape variety, excepting L and M particle sizes at 9% addition level.

As it can be seen from Figure 2 (A1-D1), the samples with GSF from white grape variety presented the higher values of the G' and G'' moduli for all particle sizes and levels of addition in wheat flour, which may be correlated with de FN index values. For GSF from red variety, different particle sizes and addition levels slightly decrease G' and

G'' values. In the gelatinization temperature range, the moduli values are lower for dough samples with GSF addition at all particle sizes for 3 to 7% addition levels probably due to the high content of fat from grape seed from red variety (Mironeasa, 2017) and due to the starch dilution. Only for GSF_9L and GSF_9M an increase of loss and storage moduli as against the control sample was recorded. The heat-induced process at the initial heating from 20 to 60°C leads to the proteins weakening, dough softening and decreases moduli values, according to the results reported by Rosell et al., (2007). An increase of both moduli can be seen once the temperature gets higher. This behaviour can be related to the starch granules which absorb the water available in the dough system during gelatinization process, increasing dough viscosity.

The results of creep-recovery measurements for the composite flour with GSF from two variety revealed similar viscoelastic behaviours to that of pure wheat dough, exhibiting an increasing strain under the applied stress and partial reformation after stress removal (Abebe et al., 2015). The creep phase comprise instantaneous elastic (1), retarded elastic (2) and viscous deformation (3), while the recovery phase, includes instantaneous elastic recovery (4) and retarded elastic recovery (5) (Figure 3). The maximum creep compliance ($J_{c_{max}}$) and the maximum recovery compliance ($J_{r_{max}}$) measured at the end of the creep and recovery phase represent the parameters readily available from the creep-recovery curves. During creep phase, the deformation reaching its maximum value, the compliance (J) increased at $J_{c_{max}}$ value at time 60 s, and then, during recovery phase, stress is removed instantly, and an appreciable decrease of compliance $J_{r_{max}}$ value was observed until almost achieved the steady state at time of 240 s (Figure 3).

From the Table 2 it is seen that GSF-wheat composite flour dough compared to the wheat flour dough showed variations in the compliance values in function of particle size, addition level and grape variety. The maximum creep compliance ($J_{c_{max}}$) values significantly decreased ($p < 0.05$) for dough samples with GSF from white variety compared to the control, the minimum values was reached for S particle size at 7% addition level, indicating higher resistances to deformation. The dough sample with low compliance resist stronger against deformation than dough with

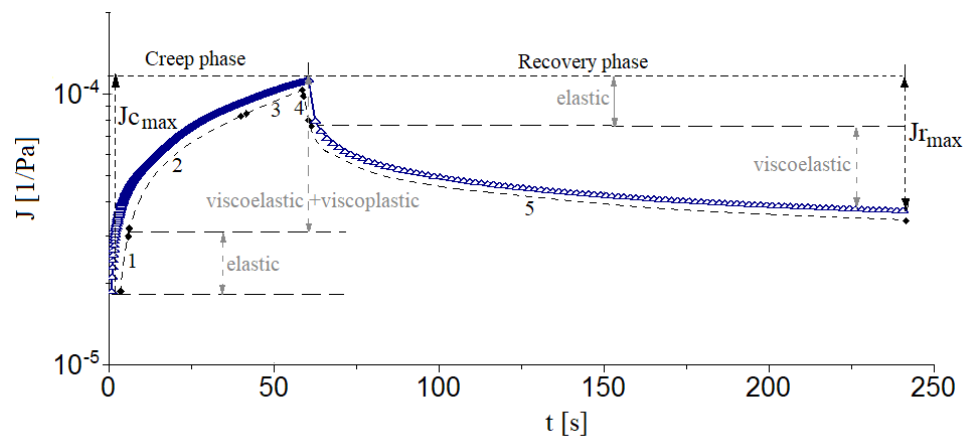


Figure 3. A typical creep-recovery curve with creep and recovery phases. The creep phase comprise instantaneous elastic (1), retarded elastic (2) and viscous deformation (3), while the recovery phase, includes instantaneous elastic recovery (4) and retarded elastic recovery (5). $J_{c_{max}}$, $J_{r_{max}}$ - maximum compliance reached in creep and recovery phase, respectively.

Table 2. The creep and recovery parameters of white wheat flour dough as influenced by grape seed flour addition from two grape varieties at different levels and particle size

Sample	GSF from		GSF from		GSF from	
	white variety	red variety	white variety	red variety	white variety	red variety
	$J_{c_{max}} \times 10^5 \text{ (Pa}^{-1}\text{)}$		$J_{r_{max}} \times 10^5 \text{ (Pa}^{-1}\text{)}$		$J_{r_{max}}/J_{c_{max}} \text{ (%)}$	
Control	17.75 ± 0.40 ^A	17.75 ± 0.40 ^A	12.20 ± 0.12 ^A	12.20 ± 0.12 ^A	68.75 ± 0.87 ^A	68.75 ± 0.87 ^A
GSF_3L	19.20 ± 1.20 ^{aBx}	18.02 ± 1.10 ^{aAx}	12.66 ± 0.80 ^{aBx}	11.61 ± 0.60 ^{bBx}	63.61 ± 0.18 ^{aBx}	64.45 ± 0.61 ^{aBx}
GSF_3M	11.31 ± 1.00 ^{aBy}	17.83 ± 0.80 ^{bAy}	7.60 ± 0.11 ^{aBy}	15.66 ± 1.10 ^{bBy}	67.49 ± 5.01 ^{aBy}	87.76 ± 2.23 ^{bBy}
GSF_3S	13.37 ± 0.90 ^{aBz}	15.62 ± 0.50 ^{bAz}	6.48 ± 0.90 ^{aBy}	12.12 ± 0.80 ^{bBz}	48.31 ± 3.49 ^{aBz}	77.54 ± 2.64 ^{bBz}
GSF_5L	13.24 ± 0.90 ^{aCx}	17.38 ± 0.90 ^{bBx}	8.05 ± 0.50 ^{aCx}	13.13 ± 0.80 ^{bAx}	60.82 ± 0.36 ^{aCx}	75.48 ± 0.69 ^{bCx}
GSF_5M	5.84 ± 0.50 ^{aCy}	14.52 ± 0.70 ^{bBy}	3.30 ± 0.40 ^{aCy}	8.45 ± 0.60 ^{bAy}	56.39 ± 2.03 ^{aCy}	58.15 ± 1.33 ^{aCy}
GSF_5S	7.78 ± 0.80 ^{aCz}	16.08 ± 0.60 ^{bBz}	6.18 ± 0.60 ^{aCy}	14.49 ± 0.60 ^{bAz}	79.47 ± 0.46 ^{aCz}	90.10 ± 0.37 ^{bCz}
GSF_7L	6.96 ± 0.80 ^{aDx}	21.00 ± 1.30 ^{bAx}	6.28 ± 0.30 ^{aDx}	16.26 ± 1.20 ^{bCx}	90.70 ± 6.16 ^{aDx}	77.39 ± 0.93 ^{bDx}
GSF_7M	6.13 ± 0.60 ^{aDy}	17.63 ± 1.00 ^{bAy}	5.46 ± 0.70 ^{aDy}	12.21 ± 0.80 ^{bCy}	88.89 ± 2.73 ^{aDy}	69.23 ± 0.61 ^{bDy}
GSF_7S	4.89 ± 0.30 ^{aDz}	16.87 ± 0.80 ^{bAz}	2.67 ± 0.40 ^{aDy}	13.75 ± 0.80 ^{bCz}	54.40 ± 4.85 ^{aDz}	81.48 ± 0.88 ^{bDz}
GSF_9L	3.87 ± 0.30 ^{aEx}	18.10 ± 0.90 ^{bCx}	3.64 ± 0.30 ^{aEx}	12.38 ± 0.70 ^{bDx}	94.03 ± 0.46 ^{aAx}	68.38 ± 0.47 ^{bEx}
GSF_9M	6.16 ± 0.50 ^{aEy}	10.95 ± 1.30 ^{bCy}	4.13 ± 0.60 ^{aEy}	9.88 ± 0.90 ^{bDy}	66.81 ± 4.33 ^{aAy}	90.43 ± 2.53 ^{bEy}
GSF_9S	7.13 ± 0.60 ^{aEz}	17.97 ± 1.40 ^{bCz}	3.75 ± 0.50 ^{aEy}	17.12 ± 1.10 ^{bDz}	52.45 ± 2.61 ^{aAz}	95.34 ± 1.31 ^{bEz}

Grape seeds flour (GSF) particle size: Large, L > 500 μm , Medium, 200 μm > M < 500 μm and Small fractions, S < 200 μm . Mean values of triplicates (\pm standard deviation) for independent evaluated samples. Mean values are followed by different superscript (a-b) in the same row (GSF from white /red variety) if are significantly different, according to Tukey's test ($p \leq 0.05$); A-E for the different addition level (3, 5, 7 and 9%) of GSF, means in the same column with different superscript were significantly ($p \leq 0.05$); x-z for the same addition level of GSF and different particle sizes (L, M and S), means in the same column with different superscript were significantly ($p \leq 0.05$).

high compliance, indicating the formation of stronger bonds between the macromolecules in wheat flour and S particle size of GSF. With GSF level increase in dough system, the maximum deformation decreased due to the increase of solid state properties, the $J_{c_{max}}$ parameter being significantly lower ($p < 0.05$) for white GSF-wheat blends than the control, except for 3% of L particle size. At the 5% level of addition, GSF from red variety conducted a lower significantly ($p < 0.05$) decrease of $J_{c_{max}}$ parameter, compared to the control (Table 2). Therefore, the dough samples with GSF from red variety had lower resistance to deformation than samples with GSF from white variety which had a strengthening effect on dough, especially for 5% level of addition of M particle size. The obtained results are in agreement with those reported by Meral and Dogan, (2013). This strengthening effect can be related to the hydroxyl groups from phenolics compounds. GSF from white variety contains higher amount of phenolics compounds than the GSF from red variety (Iuga et al., 2017). Phenolics compounds may directly interact with proteins resulting in non-covalent or covalent bonding, protein-polyphenols interactions modify proteins, influencing the quality and functional properties of food (Baxter et al., 1997). In addition, the interactions between insoluble dietary fibre present in GSF and wheat proteins may be responsible for the increase in dough stiffness, in agreement with the results reported by Goldstein et al., (2010). GSF particles can act as filler in the dough matrix and therefore, influence the resistance of dough to deformation. The decrease in dough consistency or increase in compliances is related to the dough capacity to deform. A greater resistance to deformation was reported in strong bread flour dough than softer pastry flour dough (Wang and Sun, 2002). In most baking applications bakers generally preferred strong dough due to the better rheological and handling properties, better form and texture quality of the final products.

Analysis of variance (ANOVA) results (Table 2) indicated that there was significant difference ($p < 0.05$) in $J_{c_{max}}$ GSF substituted flour dough samples with regards to both GSF varieties and all particle size. The addition level had a significant effect on $J_{c_{max}}$ parameter of dough samples with GSF from white variety. In respect to the samples with GSF from red variety, only the dough samples with 5%

and 7% addition levels differ significantly ($p < 0.05$) in $J_{c_{max}}$ compared to the control.

The recovery analysis data showed that the GSF addition in white wheat flour dough has different effect in function of grape variety, addition level and particle size. The $J_{r_{max}}$ values of the composite dough samples with GSF from white variety were significant ($p < 0.05$) lower than that of dough samples with GSF from red variety (Table 2). The doughs with GSF from red grape variety had a greater increment effect on the elastic recovery of composite flour dough than grape from white variety, especially in the case of dough samples with S particle size. An increase in elastic recovery was also obtained with increase of GSF addition level from red grape variety compared to control, which implies less deformation or breakage of the composite network (Skendi et al., 2009). A damaged structure and no links between wheat flour and GSF constituents is indicate by the small value of the elastic recovery in the case of sample with S particle size from white grape variety, except 5% addition level. But, a high value of the elastic recovery (88.89%) was found at 7% addition level for sample with M particle size. The grape variety, particle size and addition level seemed to be responsible for the variability in elastic recovery of the dough formulations. With increasing GSF addition level from white grape variety in dough sample the maximum compliance in the recovery phase ($J_{r_{max}}$) significantly ($p < 0.05$) decrease at 3, 5 and 7% addition levels than control, suggesting the irreversible breakage of the elastic bonds in the dough samples containing GSF. An increase in elastic recovery with increase of addition level was found in dough samples with GSF from red variety, compared to control (Table 2). The obtained values from recovery percentage of composite flour dough formulations are in the same range that reported values for some gluten formulations (Lazaridou et al., 2007). The ANOVA results revealed that there was significant difference ($p < 0.05$) in percentage recovery with regards to both GSF varieties and all addition levels except of 9% GSF level from white variety. The highest percentage recovery was found in dough samples with GSF from red variety. The GSF-wheat flour dough which showed high percentage recovery can resist stronger against deformation due to the formation of elastic bonds between the macromolecules in wheat flour and GSF.

Conclusion

It can be concluded that GSF incorporation in wheat flour affects both physico-chemical and rheological properties of white wheat flour dough. The moisture, ash and protein contents increased with GSF addition, the magnitude depending on the addition level, particle size and grape variety. Oscillatory and creep-recovery tests showed relevant influence of particle size, grape variety and addition level on the rheological properties. Dough samples with GSF from red grape variety exhibit a lower storage modulus in the whole range of frequency compared to the dough with GSF from white variety. Particle size and addition level have a synergistic effect on the dynamic moduli indicating the existence of some interactions between components of the GSF-wheat flour dough which modified in various way the dough matrices. In respect to the gelatinization temperatures, the samples with GSF from white grape variety presented the higher values of the G' and G'' moduli for all particle sizes and levels of addition in wheat flour. During creep-recovery tests the dough samples supplemented with small particle sizes of GSF from white grape variety showed higher resistance to deformation than those supplemented with small particle sizes from red grape variety at addition levels from 3 to 9%. It can be concluded that from the two grape varieties, the GSF from red variety can be added in a higher levels than the white one, the proper dough behaviour being obtained for small particle size. For the white grape variety, the medium particle size added up to 7% in order to get good dough behaviour is recommended.

Acknowledgments. This work was supported by a grant of the Romanian National Authority for Scientific Research and Innovation, CNCS/CCCDI – UEFISCDI, project number PN-III-P2-2.1-BG-2016-0136, within PNCDI III.

References

1. Abebe W, Ronda F, Villanueva M, Collar C, (2015). Effect of tef [*Eragrostis tef* (Zucc.) Trotter] grain flour addition on viscoelastic properties and stickiness of wheat dough matrices and bread loaf volume. *European Food Research and Technology*, 241(4): 469-478
2. Acun S, Gül H, (2013). Effects of grape pomace and grape seed flours on cookie quality. *Quality Assurance and Safety of Crops & Foods*, 6(1): 81-88
3. Addo K, Xiong Y L, Blanchard S P, (2001). Thermal and dynamic rheological properties of wheat flour fractions. *Food Research International*, 34(4): 329-335
4. Ahmed J, Al-Attar H, Arfat Y A, (2016). Effect of particle size on compositional, functional, pasting and rheological properties of commercial water chestnut flour. *Food Hydrocolloids*, 52: 888-895
5. Ahn H J, Son H S, (2012). Physicochemical properties of different grape varieties cultivated in Korea. *Korean Journal of Food Science and Technology*, 44(3): 280-286
6. Amariei S, Norocel L, Pădureț S, Gutt G, (2018). Effect of grape seed flour on the quality of summer salami. *Journal of Food Processing and Preservation*, 42(5): e13601
7. Baiano A, Terracone C, (2011). Varietal differences among the phenolic profiles and antioxidant activities of seven table grape cultivars grown in the south of Italy based on chemometrics. *Journal of Agricultural and Food Chemistry*, 59(18): 9815-9826
8. Bail S, Stuebiger G, Krist S, Unterweger H, Buchbauer G, (2008). Characterization of various grape seed oils by volatile compounds, triacylglycerol composition, total phenols and antioxidant capacity. *Food Chemistry*, 108: 1122-1132
9. Bakke A, Vickers Z, (2007). Consumer liking of refined and whole wheat breads. *Journal of Food Science*, 72(7): S473-S480
10. Balestro E A, Sandri I G, Fontana R C, (2011). Utilização de bagaço de uva com atividade antioxidante na formulação de barra de cereais. *Revista Brasileira de Produtos Agroindustriais, Campina Grande*, 13(2): 195-201
11. Baxter N J, Lilley T H, Haslam E, Williamson M P, (1997). Multiple interactions between polyphenols and a salivary proline-rich protein repeat result in complexation and precipitation. *Biochemistry*, 36: 5566-5577
12. Bender A B B, De Mello Luviélmo M, Loureiro B B, Speroni C S, Boligon A A, Da Silva L P Penna N G, (2016). Obtenção e caracterização de farinha de casca de uva e sua utilização em snack extrusado/Obtention and characterization of grape skin flour and its use in an extruded snack. *Brazilian Journal of Food Technology*, 19: 1-9
13. Bender A B B, Speroni C S, Salvador P R, Loureiro B B, Lovatto N M, Goulart F R, Lovatto M T, Miranda M Z, Silva L P, Penna N G, (2017). Grape pomace skins and the effects of its inclusion in the technological properties of muffins. *Journal of Culinary Science & Technology*, 15(2): 143-157
14. Berland S, Launay B, (1995). Rheological properties of wheat flour doughs in steady and dynamic shear: Effect of water content and some additives. *Cereal Chemistry*, 72(1): 48-52.
15. Bonnard-Ducasse M, Della Valle G, Lefebvre J, Saulnier L, (2010). Effect of wheat dietary fibres on bread dough development and rheological properties. *Journal of Cereal Science*, 52(2): 200-206
16. Brenes A, Viveros A, Chamorro S, Arija I, (2016). Use of polyphenol-rich grape by-products in monogastric nutrition. A review. *Animal Feed Science and Technology*, 211:1-17.
17. Choi Y, Choi J, Han D, Kim H, Lee M, Kim H, Lee J, Chung H, Kim C, (2010). Optimization of replacing pork back fat with grape seed oil and rice bran fibre for reduced-fat meat emulsion systems. *Meat Science*, 84: 212-218
18. Codină G G, Mironeasa S, Mironeasa C, (2012). Variability and relationship among Mixolab and Falling Number evaluation based on influence of fungal α -amylase

- addition. *Journal of the Science of Food and Agriculture*, 92(10): 2162-2170
19. Eliasson A-C, Gudmundsson M, (1996). Starch: Physico-chemical and functional aspects. In A-C Eliasson (Ed.), *Carbohydrates in food*, pp. 431–502. New York, Marcel Dekker, Chapter 10
 20. Fernandes L, Casal S, Cruz R, Pereira J A, Ramalhosa E, (2013). Seed oils of ten traditional Portuguese grape varieties with interesting chemical and antioxidant properties. *Food Research International*, 50: 161–166
 21. García-Lomillo J, Gonzalez-SanJose M L, Del Pino-García R, Rivero-Perez M D, Muniz-Rodriguez P, (2014). Antioxidant and antimicrobial properties of wine byproducts and their potential uses in the food industry. *Journal of Agricultural and Food Chemistry*, 62(52): 12595-12602
 22. Goldstein A, Ashrafi L, Seetharaman K, (2010). Effects of cellulosic fibre on physical and rheological properties of starch, gluten and wheat flour. *International Journal of Food Science & Technology*, 45: 1641–1646
 23. Guerra-Rivas C, Vieira C, Rubio B, Martínez B, Gallardo B, Mantecón A R, Lavín P, Manso T, (2016). Effects of grape pomace in growing lamb diets compared with vitamin E and grape seed extract on meat shelf life. *Meat Science*, 116: 221-229
 24. Iuga M, Ropciuc S, Mironeasa S, (2017). Antioxidant activity and total phenolic content of grape seeds and peels from Romanian varieties. *Food and Environment Safety Journal*, 16(4): 276-281
 25. Iuga M, Mironeasa S, Zaharia D, Ropciuc S, Mironeasa C, (2017). Effects of grape seeds flour addition on wheat flour dough rheological properties, SGEM Vienna Green Conference 2017, 27th – 30th November, publicat în Conference Proceeding Nano, Bio and Green-Technologies for a sustainable Future, XVII(63): 209-216
 26. Lamacchia C, Chillo S, Lamparelli S, Suriano N, La Notte E, Del Nobile M A, (2010). Amaranth, quinoa and oat doughs: mechanical and rheological behavior, polymeric protein size distribution and extractability. *Journal of Food Engineering*, 96: 97–106
 27. Lavelli V, Torri L, Zeppa G, Fiori L, Spigno G, (2016). Recovery of winemaking by-products for innovative food application. *Italian Journal of Food Science*, 28: 542-564
 28. Lazaridou A, Duta D, Papageorgiou M, Belc N, Biliaderis C G, (2007). Effects of hydrocolloids on dough rheology and bread quality parameters in gluten-free formulations. *Journal of Food Engineering*, 79(3): 1033-1047
 29. Meral R, Doğan İ S, (2013). Grape seed as a functional food ingredient in bread-making. *International Journal of Food Sciences and Nutrition*, 64(3): 372-379
 30. Mildner-Szkudlarz S, Bajerska J, Zawirska-Wojtasiak R, Górecka D, (2013). White grape pomace as a source of dietary fibre and polyphenols and its effect on physical and nutraceutical characteristics of wheat biscuits. *Journal of the Science of Food and Agriculture*, 93(2): 389-395
 31. Mironeasa S, (2017). Valorisation of secondary products from wine making, Publishing House Performantica, Iasi, Romania.
 32. Mironeasa S, Iuga M, Zaharia D, Dabija A, Mironeasa C, (2017). Influence of particle sizes and addition level of grape seeds on wheat flour dough rheological properties, SGEM Vienna Green Conference 2017, 27th – 30th November, publicat în Conference Proceeding Nano, Bio and Green-Technologies for a sustainable Future, XVII(63): 265-272
 33. Mironeasa S, Codină G G, Mironeasa C, (2016). Optimization of wheat-grape seed composite flour to improve alpha-amylase activity and dough rheological behavior. *International Journal of Food Properties*, 19(4): 859-872
 34. Mironeasa S, Codină G G, Mironeasa C, (2012). The effects of wheat flour substitution with grape seed flour on the rheological parameters of the dough assessed by Mixolab. *Journal of Texture Studies*, 43: 40–48
 35. Mironeasa S, Mironeasa C, Codină G G, (2010). Evaluation of mineral element content in grape seed and defatted grape seed. *Food and Environment Safety*, 53-60
 36. Mohammed I, Ahmed A R, Senge B, (2011). Dynamic rheological properties of chickpea and wheat flour dough's. *Journal of Applied Sciences*, 11(19): 3405-3412
 37. Moreira R, Chenlo F, Torres M D, Prieto D M, (2010). Influence of the particle size on the rheological behaviour of chestnut flour doughs. *Journal of Food Engineering*, 100(2): 270-277
 38. Piovesana A, Bueno M M, Klajn V M, (2013). Elaboração e aceitabilidade de biscoitos enriquecidos com aveia e farinha de bagaço de uva/Elaboration and acceptability of cookies enhanced with oat and flour grape pomace. *Brazilian Journal of Food Technology*, 16(1): 68-72
 39. Rockenbach I I, Jungfer E, Ritter C, Santiago-Schübel B, Thiele B, Fett R, Galensa R, (2012). Characterization of flavan-3-ols in seeds of grape pomace by CE, HPLC-DAD-MSn and LC-ESI-FTICR-MS. *Food Research International*, 48(2): 848-855
 40. Rosell C M, Collar C, Haros M, (2007). Assessment of hydrocolloid effects on the thermo-mechanical properties of wheat using the Mixolab. *Food Hydrocolloids*, 21: 454–462
 41. Salvador A, Sanz T, Fiszman S M, (2006). Dynamic rheological characteristics of wheat flour–water doughs. Effect of adding NaCl, sucrose and yeast. *Food Hydrocolloids*, 20(6): 780-786
 42. Saura-Calixto F, (1998). Antioxidant dietary fiber product: a new concept and a potential food ingredient. *Journal of Agricultural and Food Chemistry*, 46(10): 4303-4306
 43. Scalbert A, Manach C, Morand C, Rémésy C, Jiménez L, (2005). Dietary polyphenols and the prevention of diseases. *Critical Reviews in Food Science and Nutrition*, 45(4): 287-306
 44. Skendi A, Papageorgiou M, Biliaderis C G, (2009). Effect of barley b-glucan molecular size and level on wheat dough rheological properties. *Journal of Food Engineering*, 91: 594–601
 45. Sundarram A, Murthy T P K, (2014). α -amylase production and applications: a review. *Journal of Applied & Environmental Microbiology*, 2(4): 166-175
 46. Wang F C, Sun X S, (2002). Creep recovery of wheat flour doughs and relationship to other physical dough tests and bread making performance. *Cereal Chemistry*, 79: 567–571
 47. Weipert D, (1990). The benefits of basic rheometry in studying dough rheology. *Cereal Chemistry*, 67(4): 311-317