

The Role of Formulation and Working Parameters on the Rheological Properties of Semolina Doughs for the Production of *Carasau* Bread

Nicola Melis^a, Fabio Fanari^b, Francesco Desogus^{a*}, Massimiliano Grosso^a

^aDipartimento di Ingegneria Meccanica, Chimica e dei Materiali (DIMCM), Università degli Studi di Cagliari, Via Marengo 2, 09123, Cagliari, Italy;

^bFood Safety and Functionality Programme, Institute of Agriculture and Food Research and Technology (IRTA), 17121 Monells, Spain;
francesco.desogus2@unica.it

Carasau bread is a typical Sardinian baking product, with great commercial potential, due to its long shelf life. Nowadays, its production is performed, in most cases, in small or medium size factories, where the working conditions and quality properties of the product are set on an empirical basis. Thus, the processing know-how lacks quantitative information, and the product is still far from standardization. As a result, industrial-scale manufacturing is hindered. The literature presents some studies devoted to better explaining the effect of semolina doughs' main constituents (gluten, starch, etc.) on their rheological properties or to infer the latter through in-line measurement. However, it is still necessary to understand the role of each working parameter in conditioning the dough rheology.

This work investigated the role of five working parameters: yeast amount, salt amount, water temperature, kneading time, and leavening time. The water amount was kept constant to avoid covering other effects because its role can be predominant in most cases. A Design of Experiments (DOE) was performed, in order to plan the experimental campaign. First, the dough samples were tested through a parallel plate rheometer (Anton Paar, model MCR 102), applying both creep and frequency sweep tests. Moreover, the same samples were subjected to Texture Profile Analysis (TPA) to highlight possible correlations between theoretical rheological model parameters and TPA ones, which can be obtained in shorter times, so being more suitable for process monitoring purposes.

1. Introduction

Carasau bread is a traditional Sardinian flatbread made from re-milled durum wheat semolina, water, yeast, and salt. This kind of bread is characterized by its crispiness due to the very thin sheets in which it is produced (less than 1 mm thick) and by its long shelf-life with respect to other classic bread due to its very low moisture content. Such features make it a very appealing and unique product with great commercial potential, whose demand is increasingly growing in the Italian market, the rest of Europe, and the world (Pagani et al., 2014). Despite the expanding market, the production of *Carasau* bread is mainly carried out in a small or medium scale semi-artisanal way. Controlled industrial production has some drawbacks, largely related to the traditional production process which is based on empirical monitoring and the knowledge and experience of the operators (Baire et al., 2021). The industrial production of this kind of bread to meet the market demand without compromising the high quality that characterizes the traditional process is still a challenge that requires a high degree of bakeries automation and real-time control of the entire production process (Mannaro et al., 2022; Baire et al., 2018). Dough's microstructure and texture are of fundamental importance because their mechanical properties affect the final product's quality and characteristics. These aspects are the outcome of both the formulation and the kneading process that lead to the formation of the gluten network, a three-dimensional structure (Fanari et al., 2019b; Fanari et al., 2022a). As a consequence of this consideration, the rheological characterization of the dough and the understanding of how the dough's rheological properties – and, hence, the final product ones –

correlate with the main working parameters are probably the most appealing way to gain significative and direct insights on the on-going process of production (Menjivar, 1990).

In this context, the key role of the ingredients concerning their interaction with the gluten proteins has already been pointed out in the literature (Fanari et al., 2019a; Fanari et al., 2020a), as well as the importance of their relative amount because they all compete for binding water (Mani et al., 1992). Thus, among all the ingredients, water has probably the most predominant role, often hiding the effects of the other components and making the rationalization of the whole process difficult to understand. The role of salt (sodium chloride) has been well established. Indeed, apart from the involvement in the overall organoleptic properties, it deeply affects all the production steps (Miller and Hosenev, 2008). In the formation of the dough, salt plays a key role in water distribution (Fanari et al., 2020b), strongly affecting the final elasticity (Bloksma, 1990) and delaying the formation of the gluten network due to a reduction in the hydration rate of the gluten (McCann and Day, 2013). It also leads to a higher mixing resistance of the dough and a reduced stickiness, and it also stabilizes the yeast fermentation rate, other than other post-production features such as the inhibition of microbial growth of stored bread (Chen et al., 2019). The role of yeast is mainly related to the leavening process and, therefore, to the fermenting agent's CO₂ production within the dough bulk (Lodi et al., 2021). Ethanol, acetic and succinic acids are some other fermentation products which influence the organoleptic characteristic of the baked product and its mechanical properties. Indeed, as reported by Meerts et al. (2018), they are involved in forming the gluten network, and modifying its structure effectively.

From a purely rheological point of view, the effects of many production parameters on the final properties of doughs have been described in the literature with non-uniform or contrasting results (Fanari et al., 2022b). For example, this is the case with the salt effect on the storage modulus (G'). A general consensus is that an increase in salt content leads to an increase in storage modulus, as reported, among others, by Beck et al. (2012) and Chen et al. (2019). On the other hand, Lynch et al. (2009) did not observe a significant reduction of G' with decreasing the salt amount; nevertheless, the complete absence of salt led to a decrease in the elastic module. Regarding yeast's effect, rheological studies on fermented doughs are limited, probably due to the experimental difficulties in studying materials with characteristics changing over time.

This work investigated the single and combined effect of five working parameters on the rheological properties: salt amount, yeast amount, water temperature, mixing time and leavening time. As already pointed out, despite the importance of the water amount, this parameter was kept constant to outline better, or avoid hiding, the influence of the other variables. Furthermore, the experimental campaign was planned using the Design of Experiments (DOE) technique described further in this paper.

2. Rheology of doughs: models

The dough is known to possess a viscoelastic, non-linear behaviour, so the stress is a function of both the applied strain and the strain rate (Faridi and Faubion, 2012). This dependency is studied through dynamic oscillation measurement, particularly small amplitude oscillatory shear (SAOS) tests, which usually operate in the linear viscoelastic deformation regime with low strain values (Morrison, 2001). The dependency of the viscoelastic module with the strain frequency for semolina doughs is well described by the "weak gel model" (Gabriele et al., 2001), which considers the presence of a supramolecular structure formed by droplets, solid particles, fibres, etc. According to this theory, the internal organization of this superstructure by means of weak intramolecular interactions has the viscoelastic behaviour of the material as a consequence. In fact, the material mainly behaves as a solid ("strong gel") with low stresses. On the other hand, it acts as a liquid when high stresses are applied, and the weak interactions break. Dependence of storage and loss moduli, G' and G'', on the frequency are modelled with a power law model (Bohlin and Carlson, 1981), as reported in Eq(1) and Eq(2).

$$G'(\omega) = K_0' \omega^{n'} \quad (1)$$

$$G''(\omega) = K_0'' \omega^{n''} \quad (2)$$

Doughs show a non-linear response also towards deformation, as they can partially recover their initial structure. The compliance J , defined as the ratio between the strain $\varepsilon(t)$ and the constant stress σ_0 , is the usual choice for quantifying creep results. This parameter is usually described as a function of time by mechanical models describing elements such as springs and dampers to explain this rheological behaviour. The Burgers model (Mainardi and Spada, 2011), one of the most used ones, includes four parameters, as follows:

$$J(t) = J_0 + J_m \cdot \left(1 - e^{-\frac{t}{\tau}}\right) + \frac{t}{\eta_0} \quad (3)$$

In this model, the compliance J is described as a function of time t , with the instantaneous and delayed compliance, respectively J_0 and J_m , the delay time τ , and the material's static viscosity η_0 . The viscoelastic

properties of doughs can also be evaluated through texture analysis, consisting of operating axial compressions at a fixed speed of the probe and measuring the force exerted by the sample. In particular, such tests were performed according to Texture Profile Analysis (TPA), and the following parameters were obtained, according to Peleg (2019): hardness (Ha), cohesiveness (Co), elasticity (EI), gumminess (Gu) and toughness (To).

3. Materials and methods

Dough samples were prepared with commercial re-milled semolina (Brundu, carbohydrates 71.0 %_{wt}, proteins 13.0 %_{wt}, fat 1.5 %_{wt}), commercial fresh brewer's yeast (*Saccharomyces cerevisiae*), commercial sea salt and distilled water. Each sample was prepared in a bench kneader, mixing 500 g of semolina and 260 g of water (52 %_{wt}). The factors chosen to vary were salt amount (m_s), yeast amount (m_y), water temperature (T_w), mixing time (t_{mix}), and leavening time (t_{leav}). A design of experiment (DOE) protocol consisting of two levels, five factors and half fraction of the full factorial, with resolution V, was used to reduce the number of experiments (Montgomery, 2013). In fact, due to the long-term duration of the experiments, the fractional factorial design was an obliged choice to describe the process under investigation at reasonable times. Table 1 reports the levels adopted for each factor varied.

Table 1: Levels at low and high values

Factor/Level	Low	High	Units
A = Salt	1.0	2.0	%
B = Yeast	1.0	2.0	%
C = Water Temperature	20	28	°C
D = Mixing Time	15	25	min
E = Leavening Time	20	40	min

Three center points were further considered to check the model's linearity. In the end, 35 different experimental conditions were explored and replicated twice. Finally, the fractional factorial design was created using the generator E=ABCD. Table 2 reports the details of the DOE, together with the complete alias structure. Notice that single effects and two-way interactions can be resolved from higher-order interactions because of the resolution V of the chosen fractional factorial design.

Table 2: Generators, defining relations and complete alias structure

Terms			
Generator	E=ABCD		
Defining relation	I=ABCDE		
Alias Structure	A+BCDE	B+ACDE	C+ABDE
	D+ABCE	E+ABCD	AB+CDE
	AC+BDE	AD+BCE	AE+BCD
	BC+ADE	BD+ACE	BE+ACD
	CD+ABE	CE+ABD	CD+ABC

The rheological measurements were performed using an MCR 102 Anton-Paar rheometer (Anton Paar GmbH, Austria), with a parallel plate geometry equipped with a 25 mm plate and a 2 mm gap. The measurement temperature in the rheometer was kept constant at 25 °C using a heating system based on the Peltier technology. All the measurements were carried out both right before and right after the leavening process. Frequency sweep tests were performed with frequency ranging from 1 to 100 rad·s⁻¹ with a constant strain of 0.1 %, which was the estimated linear viscoelastic limit (LVE) according to preliminary amplitude sweep tests. Complex modulus (G^*), storage modulus (G') and loss modulus (G'') data obtained from these experiments were modelled as a function of the frequency by means of the power law model, computing the values of the model parameters (K_0 and n).

Creep measurements were conducted by applying to the sample a 50 Pa constant stress for 120 s. Compliance data obtained from these experiments were modelled as a function of time using the Burgers model, Eq(3), and the values of the model parameters (J_0 , J_m , τ , and η_0) were calculated.

TPA tests were performed with a homemade device consisting of a cylindrical probe with a 3.5 cm diameter and a cylindrical sample holder with 6.4 cm internal diameter and containing samples of 2.6 cm height. Each element of the compression tool (see Figure 1) was produced in PLA by 3D printing. The proposed fixture can perform a typical TPA measurement, and the experimental test can be conducted with the same rheometer and on the same samples as before. Analyzing and acquiring data can be then achieved by utilizing the Anton Paar software. The penetration depth was 7 mm and it was reached twice at a constant speed of 1 mm s^{-1} .

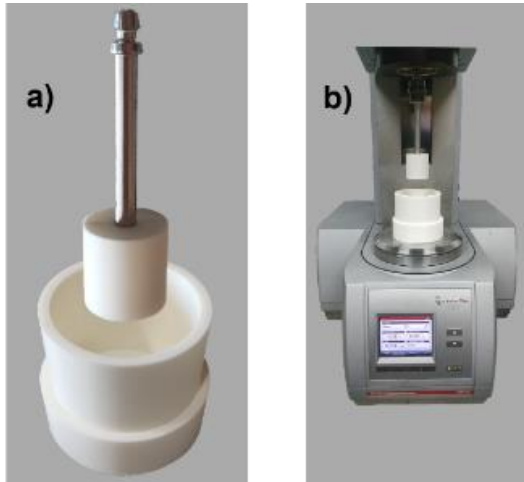


Figure 1: (a) Fixture designed to mimic the Texture Process Analyser; (b) Setup of the fixture onto the rheometer

4. Results and discussion

Regarding the data analysis for the dough samples collected before the leavening process, the dependence on the factors has been evaluated by means of a linear model for each estimated rheological parameter. The factors significantly affecting the response are selected by performing a backward elimination with a threshold p-value equal to 0.05. Results of the procedure are reported in Table 3, in terms of positive (+) and negative (-) interactions. In addition, the determination coefficients R^2 for the different linear models are also reported.

Table 3: Rheological parameters of unleavened dough samples

	K_0^*	n^*	K_0'	n'	K_0''	n''	J_0	J_m	τ	η_0	Ha	Co	El	Gu	To
m_s						+			+	-					
m_y	-		-			-				-	+	+		+	
T_w									-	-		+		+	+
t_{mix}	-	+	-	+						-	+		+		+
$m_s * m_y$										+					
$m_s * T_w$									+						
$m_s * t_{mix}$															
$m_y * T_w$												-		-	
$m_y * t_{mix}$	+		+								+				
$T_w * t_{mix}$															
R^2	0.22	0.15	0.20	0.11		0.30				0.34	0.21	0.14	0.12	0.18	0.16

Results show a strong dependency on the rheological behaviour of doughs by the mixing time. Such interaction mainly affects the complex's model parameter n and storage moduli. The loss modulus, which seems unaffected by the mixing time, shows a strong interaction between n'' and m_s . These effects may be related to gluten network formation, where the role of salt has been highlighted and which logically affects the mixing process. Interestingly, the two parameters influence two different components of the complex modulus. The yeast amount plays a role in combination with t_{mix} , which is reasonable since leavening would start from the formation of the dough. This combination affects K_0^* , K_0' and the hardness. Other strong positive interactions exist, involving the elasticity with t_{mix} , following the same effect on n^* and n' , and the amount of salt in combination with m_y and T_w , with τ and η_0 , respectively.

From testing leavened samples, t_{mix} confirmed a predominant role, influencing most parameters. The same key role is played by t_{leav} . Both variables showed a strong influence on n of complex and storage moduli, as well as on J_0 , J_m and on elasticity. Salt and yeast amounts strongly affect the loss modulus (n'' and K_0'' , respectively). The same dependency of n'' on m_s and m_y has already been observed in unleavened samples. Moreover, t_{leav} also influences the complex and storage moduli, in combination with T_w and t_{mix} . More precisely, $T_w * t_{leav}$ strongly affects the K_0' , while $t_{mix} * t_{leav}$ affects n .

Table 4: Rheological parameters of leavened dough samples

	K_0'	n'	K_0''	n''	K_0'''	n'''	J_0	J_m	τ	η_0	Ha	Co	El	Gu	To
m_s						+				+		+	+	+	+
m_y					+	-		-				+	+	+	-
T_w		-		-									+		
t_{mix}	-	+	-	+		+	+	+		-	-	+	+		
t_{leav}	-	+	-	+		+	+	+	+		-	-	+		
$m_s * m_y$															
$m_s * T_w$												-		-	-
$m_s * t_{mix}$													-		
$m_s * t_{leav}$										-					
$m_y * T_w$															
$m_y * t_{mix}$															
$m_y * t_{leav}$															
$T_w * t_{mix}$															
$T_w * t_{leav}$		+		+											
$t_{mix} * t_{leav}$	+	-	+	-			-	-			+	-			
R^2	0.30	0.57	0.30	0.58	0.16	0.60	0.34	0.47	0.22	0.24	0.21	0.49	0.74	0.32	0.29

5. Conclusions

The rheological properties of semolina dough are influenced by its formulation and production conditions in a complex way. Therefore, not only are single factors relevant but also some combinations of them. In the explored range of values, the mixing and the leavening time were the most significant parameters, which is congruent with the formation of the gluten network and the gas inclusions in the leavened dough. Nonetheless, other parameters also had an impact on the dough characteristics, such as the elasticity, which is affected by all the experimental parameters. It is expected that the results of this study will provide a preliminary indication as to which aspects need to be explored in more detail with regard to the expectations for bread quality and the optimization of industrial production processes.

Acknowledgements

This research was funded by Italian Government (Ministero dello Sviluppo Economico), Fondo per la Crescita Sostenibile – Sportello "Agrifood" PON I&C 2014–2020, Prog. n. F/200133/01–03/X45. We acknowledge Dr. Andrea Melis (University of Cagliari) for his support in 3D printing.

References

- Baire M., Melis A., Lodi M.B., Fanti A., Mazzarella G., 2018, Study and design of a wireless sensors network for the optimization of bread manufacturing process, 26th Telecommunications Forum, TELFOR 2018 - Proceedings, art. no. 8612060.
- Baire M., Melis A., Lodi M.B., Lodi L., Fanti A., Mazzarella G., 2021, Empowering traditional carasau bread production using wireless sensor network, Proceedings - IEEE International Symposium on Circuits and Systems, no. 9401216.
- Beck M., Jekle M., Becker T., 2012, Impact of sodium chloride on wheat flour dough for yeast-leavened products. I. Rheological attributes, Journal of the Science of Food and Agriculture, 92 (3), 585–592.
- Bloksma A.H., 1990, Rheology of the breadmaking process, Cereal Foods World, 2 (35), 228–236.
- Bohlin L., Carlson T.L.G., 1981, Dynamic viscoelastic properties of wheat flour dough: dependence on mixing time, Cereal Chemistry, 57 (3), 174–177.
- Chen G., Ehmke L., Sharma C., Miller R., Faa P., Smith G., Li Y., 2019, Physicochemical properties and gluten structures of hard wheat flour doughs as affected by salt, Food Chemistry, 275, 569–576.

- Fanari F., Carboni G., Grosso M., Desogus F., 2019a, Thermogravimetric analysis of different semolina doughs: effect of mixing time and gluten content, *Chemical Engineering Transactions*, 75, 343–348.
- Fanari F., Desogus F., Scano E.A., Carboni G., Grosso M., 2019b, The rheological properties of semolina doughs: influence of the relative amount of ingredients, *Chemical Engineering Transactions*, 76, pp. 703–708.
- Fanari F., Desogus F., Scano E.A., Carboni G., Grosso M., 2020a, The effect of the relative amount of ingredients on the rheological properties of semolina doughs, *Sustainability*, 12 (7), 2705.
- Fanari F., Carboni G., Grosso M., Desogus F., 2020b, Thermal properties of semolina doughs with different relative amount of ingredients, *Sustainability*, 12 (6), 2235.
- Fanari F., Carboni G., Desogus F., Grosso M., Wilhelm M., 2022a, A chemometric approach to assess the rheological properties of durum wheat dough by indirect FTIR measurements, *Food and Bioprocess Technology*, 15 (5), 1040–1054.
- Fanari F., Iacob C., Carboni G., Desogus F., Grosso M., Wilhelm M., 2022b, Broadband Dielectric Spectroscopy (BDS) investigation of molecular relaxations in durum wheat dough at low temperatures and their relationship with rheological properties, *LWT*, 161, no. 113345.
- Faridi H., Faubion J.M., 2012, *Dough Rheology and Baked Product Texture*, Springer Science & Business Media, Boston, USA.
- Gabriele D., de Cindio B., D'Antona P., 2001, A weak gel model for foods, *Rheologica Acta*, 40 (2), 120–127.
- Lodi M.B., Curreli N., Melis A., Garau E., Fanari F., Fedeli A., Randazzo A., Mazzarella G., Fanti A., 2021, Microwave characterization and modeling of the carasau bread doughs during leavening, *IEEE Access*, 9, 159833-159847.
- Lynch E.J., Dal Bello F., Sheehan E.M., Cashman K.D., Arendt E.K., 2009, Fundamental studies on the reduction of salt on dough and bread characteristics, *Food Research International*, 42 (7), 885–891.
- Mainardi F., Spada G., 2011, Creep, relaxation and viscosity properties for basic fractional models in rheology, *The European Physical Journal Special Topics*, 193 (1), 133–160.
- Mani K., Trägårdh C., Eliasson A.-C., Lindahl L., 1992, Water content, water soluble fraction, and mixing affect fundamental rheological properties of wheat flour doughs, *Journal of Food Science*, 57 (5), 1198–1209.
- Mannaro K., Baire M., Fanti A., Lodi M.B., Didaci L., Fedeli A., Cocco L., Randazzo A., Mazzarella G., Fumera, G., 2022, A robust SVM color-based food segmentation algorithm for the production process of a traditional carasau bread, *IEEE Access*, 10, 15359-15377.
- McCann T.H., Day L., 2013, Effect of sodium chloride on gluten network formation, dough microstructure and rheology in relation to breadmaking, *Journal of Cereal Science*, 57 (3), 444–452.
- Meerts M., Ramirez Cervera A., Struyf N., Cardinaels R., Courtin C.M., Moldenaers P., 2018, The effects of yeast metabolites on the rheological behaviour of the dough matrix in fermented wheat flour dough, *Journal of Cereal Science*, 82, 183–189.
- Menjivar J.A., 1990, Fundamental aspects of dough rheology, In: *Dough rheology and baked product texture*, Springer US, Boston, USA, 1 – 28.
- Miller R., Hosenev R., 2008, Role of salt in baking, *Cereal Foods World*, 53, 4-6
- Montgomery D.C., 2013, *Design and Analysis of Experiments*, 8th ed., John Wiley & Sons, Inc.
- Morrison F.A., 2001, *Understanding Rheology (Topics in Chemical Engineering)*, Oxford University Press, New York, USA.
- Pagani M.A., Lucisano M., Mariotti M., 2014, Italian bakery products, In: *Bakery products science and technology*, 2nd ed., John Wiley & Sons, Ltd
- Peleg M., 2019, The instrumental texture profile analysis revisited, *Journal of Texture Studies*, 50 (5), 362–368.