

Rheology of wheat flour dough at mixing

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▶ To cite this version:

Guy Della Valle, Hubert Chiron, Luc Saulnier, Maude Dufour, Florence Hugon, et al.. Rheology of wheat flour dough at mixing. Current Opinion in Food Science, 2022, 47, pp.100873. 10.1016/j.cofs.2022.100873. hal-03714931

HAL Id: hal-03714931 https://hal.inrae.fr/hal-03714931v1

Submitted on 22 Jul 2024

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1 Rheology of wheat flour dough at mixing 2 Guy Della Valle^{1*}, Maude Dufour^{1,2}, Florence Hugon², Hubert Chiron¹, Luc Saulnier¹, Kamal Kansou¹ 3 4 ¹ INRAE UR-1268 Biopolymères Interactions et Assemblages, 44316 Nantes, France 5 ²La Boulangere & Co, 85140 Essarts en Bocage, France 6 7 * corresponding author: guy.della-valle@inrae.fr 8 9 **Abstract** 10 The baking industry performs common technological (empirical) tests. Unfortunately, the results 11 hardly predict the dough behaviour on-line. This paper first reviews the most common methods to 12 assess the rheological properties of the dough, including empirical tests, small and large 13 deformations methods. A second section describes the relations between rheological properties and 14 dough behaviour at the critical step of dough mixing. We put forward a tentative interpretation of 15 these relations based on dough structural changes supported by results from imaging or 16 spectroscopic methods. Finally, a review of simple models consistent with the physical understanding 17 of the dough behaviour is presented as tools that scientist and engineers can use to interpret 18 experimental data, perform system analysis and anticipate the product properties. 19 20

Introduction

Anticipating the mechanical behaviour of the wheat flour dough is a major issue for the baking industry. With the increasing variability of the wheat quality, due to environmental changes or to sourcing diversification, it becomes essential to comprehensively assess the common rheological models in a review with a view to examine their capacity to support decision in industry. Interestingly, most rheological properties of the dough are determined as soon as the mixing operation, the first operation of the processes. The mixing brings the mechanical energy and the deformation required to distribute and hydrate homogenously the flour constituents and to form the network of gluten proteins. The gluten network is largely responsible for the visco-elastic properties of the dough, its role in the retention and the stabilization of the air cells in the dough is well documented [1,2]. A non-negligible amount of air (air fraction or porosity ≥ 0.1) is actually trapped during mixing [1], which conditions the fermentation and eventually many quality criteria of bakery products. This includes nutritional properties as well, such as Glycaemic Index and dietary fiber content [2, 3], also strongly dependent of the dough rheology at mixing.

The present review offers a concise overview of the advances in dough rheology techniques and models and propose guidelines for improving suitability to issues of the baking industry.

Dough rheological properties

Many rheological tests can be performed on dough. They can be classified according to the strain mode implemented (shear or extension), the variable imposed (strain or stress) and its variation with time (dynamic or oscillatory, or steady).

Empirical testing

Due to the essential role of rheological properties in the evaluation of the technological quality of a flour, and therefore of its commercial value, manufacturers have developed empirical measurement methods, to mimic processing conditions [4]. The Farinograph® measures the torque developed during kneading in order to draw a consistency curve which presents a peak indicating the optimum development of the dough; the Extensograph® measures the force required to stretch a dough cylinder down its middle to determine its resistance; the Rheofermentometer® follows the gas release of a dough under stress, and thus predicts its ability to expand during fermentation; the Alveograph® measures the pressure required to break a film of dough, the area under the recorded pressure curve would be related to the "force" of the flour. Clearly, this test is among the most

popular among professionals, and still receives scientific attention, e.g. for determining the technological value of the wheat varieties [5]. Although these methods may be reproducible when conveniently handled, their results, generally expressed in arbitrary units, can only be used to perform relative comparison of a set of flours and the test conditions are generally insufficient to support a physical interpretation based on the flour composition or the dough structure. Indeed, they involve different modes of deformation simultaneously in an uncontrolled way, which prevents the relevant determination of intrinsic rheological properties in relation to the dough structure.

Small deformations ("fundamental rheology")

Dynamic shear measurements are carried out at low strains (≤ 0.1% for wheat flour doughs), i.e. in the linear viscoelastic domain (LVD). In practice, they require a few grams of dough prepared in small laboratory kneaders, such as the Mixograph®, where the continuous torque measurement can detect peak formation, which reflects the optimum consistency of the dough [6]. These testing conditions are far from those encountered in the process, and do not generally lead to good correlations between the results, i.e. dough viscoelastic properties, with flour processing performances [7]. Nevertheless, they have shown that the dough behaves more like a solid than a liquid (G '> G' '), and highlighted the role of high molecular weight glutenins (HMWG) and their entanglements in the creation of the gluten network [8], whilst dough viscoelastic behavior could be represented by a weak gel power law model, the exponent of which reflects the network connectivity [9]. At higher strain, beyond LVD, large amplitude oscillatory shear tests applied to gluten gels, combined with this mathematical model, show that network connectivity decreases and evidence segments extensibility [10]. However, starch/gluten blends fail to reproduce the viscoelastic behavior of flour doughs [11, 12], which indirectly highlights the role of minor constituents, such as the polysaccharides from cell wall components (CWC).

In the linear domain as well, the dynamic thermo-mechanical analysis (DMA), simulates the behavior of dough in compression or in tension during baking, by following variations of the storage module E 'with temperature. The storage modulus E '(T) curve displays a minimum value (E'min) around T=50 °C and a maximum value (E'max) close to T= 75 °C. The larger the ratio (E'max / E'min), the more the gluten network crosslinking of the dough and, to a lesser extent, the starch granules swelling, between 50 and 75°C [13, 14].

Large deformations

Measurements performed in large deformations can be associated to the behavior of the dough during processing. The shear viscosity η ($\dot{\gamma}$) can be measured by capillary rheometry, provided that

the pressure fluctuations, linked to the slippage and dough structure sensitivity, are controlled and reduced [15, 16]. In rotational rheometry, the application of creep tests (with grooved plates), by imposing various stresses, allows determining a flow curve η ($\dot{\gamma}$) [12, 17, 18], that can be fitted by the Cross model:

$$\eta = \eta_0 / (1 + (\dot{\gamma} / \dot{\gamma}_0)^n) \tag{1}$$

where η_0 is the plateau (Newtonian) viscosity; at higher shear rate, viscosity decreases according to a power law with a slope - n; the transition between the two regimes may be identified by the value of $\dot{\gamma_0}$ (s⁻¹), the reverse of which can be considered as a relaxation time of the dough, of one hour as an order of magnitude.

Extension is involved in many food processes and it induces a deformation that implies the deformation of the structural entities (molecules, aggregates, droplets, etc) along the streamlines [19]. The ring stretch test, popularized as Kieffer test, provides a measurement of the uniaxial elongational viscosity [20], while the Extensional Viscosity Fixture is used to characterize the dough strain-hardening behaviour with a better control of strain [21]. The bi-extensional, or biaxial elongation, viscosity, η_e , is the property by which the dough resists the growth of gas cells during fermentation, highlighting its strain-hardening behavior, and reflecting its capacity for gas retention and stability of the dough at rest [22]. η_e can be determined by a uniaxial compression of the dough between lubricated plates (Lubricated Squeezing Flow, LSF) [23, 24]. In the case of dough made from cereal flours, the measurements of the stress σ , of the equi-biaxial strain ε_e , and of the strain rate ε_e , lead to the determination of η_e according to the following equation [25, 26]:

$$\sigma = \eta_e * \dot{\varepsilon}_e = K (\dot{\varepsilon}_e)^m * \exp(SHI^* \varepsilon_e)$$
 (2)

where K and m are the consistency and flow indices, respectively, defined at constant strain value, SHI being the shear hardening index (> 1). Note that the consistency index K should not be confounded with the consistency derived from torque curves recorded on instrumented mixers (Farinograph, Mixograph, etc). Although dough behaves like a solid rather than a fluid, it never reaches a true state of equilibrium. However, the comparison of the results of LSF tests carried out at constant displacement speed with those obtained at constant strain rate [27], shows that, at constant strain, η_e can be expressed by a power law as deduced from equation (2). Bi-extensional properties can also be determined by the bubble inflation test [28]. The results of this test can be related to those of the Chopin Alveograph [29, 30], provided the accurate measurement of the dough film thickness [31], and $\epsilon_e \le 1.5$, which actually holds for the proofing stage [32]. The same group has determined the shear and extension behaviours, inc. the use of LSF, and proposed a composite

micromechanical model that evidenced the decohesion between gluten network and starch grains [33]. Note that mathematical expressions of stress, modulus or viscosity, as function of strain, and strain rate, which are simply illustrated by eq.1-2, are mathematical rheological models, also called constitutive equations (see Figure 1). More accurate mathematical models may be found in the literature, especially to take into account small and large strains, shear and elongational deformations, and time dependence. Finally, rheological tests are mostly performed on unyeasted dough, although fermentation and metabolites have been shown to reduce slightly extensibility and extensional viscosity [34, 35], which was attributed to gluten network degradation [36].

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Dough mixing follow-up

Various mixer geometries and working conditions affect bread dough readiness, delineated by adequate mixing time and level of water added [37]. Dough readiness is usually assessed by an optimum consistency target at mixing with the Farinograph, which is a sound and practical indicator for the baker. However, it remains difficult to relate this property with rheological properties, although some indicators, such as maximum values of SHI or of extensional stress, are inferred for the optimum dough development [38, 39]. The way the different devices impart strain to the dough samples induces distinct mechanical treatments which likely clouds the relations between the results [40]. The dough development along mixing is followed by the measurement of the power (or torque) curve P(t), and of the Specific Mechanical Energy (SME) deduced from the area under the curve (Figure 2). For wheat, values of SME for an optimum dough development generally stand in the interval [20-60 kJ.kg⁻¹] [39, 41]. The negative correlation between the SME and storage ratio measured by DMA shows that SME contributes to the creation of the gluten network [41]. This relation is further specified through the decoupling of the mechanical and kinetic effects on the unextractable fraction of glutenin polymers [42]. The optimum SME value depends on the type of deformation mode which results from the mixer geometry. Indeed, with a z-blade and planetary mixers, shear and extensional deformations contribute in a distinct manner to the development of dough structure [41, 43]. By analogy with fluid mixing principles [42, 44], whatever the mixer geometry, the shear viscosity η can be evaluated through power measurements:

145 SME /
$$(t_m * \rho_d) = \eta * (A_m. \dot{\gamma})^2$$
 (3)

 t_m being the mixing time and ρ_d the density of the dough (\approx 1.2 kg.L⁻¹ [3]), A_m is a constant, characteristic of the mixer geometry linking $\dot{\gamma}$ to the rotation speed of the mixer arm. The flow curve

of a dough can thus be used to determine A_m (= 1.55 for a z-mixer with a capacity of 5 kg) [41]). A_m values can be used to compare the performance of different mixers, in particular their capacity to dissipate (shear) or store (elongation) energy in the dough, the latter being more favorable to dough structuring, whereas the former leads to premature degradation of the gluten network [45, 46]. In addition, the spreading of the P(t) curve was found to be infuenced by dough extensibility [47]. Besides its use for rheometry, P(t) can monitor dough changes, for instance when incorporating ingredients to the flour such as insoluble wheat components (CWC) to increase the dietary fiber content, a huge challenge in baking industry. When CWC are added, SME increases, and so does the dough consistency index (K in Eq. (2)), while the ratio (E'max / E'min) measured by DMA decreases [48]. The increase of the dough consistency index K is unlikely to be the result of a higher gluten network structuring since CWC can inhibit the creation of S-S bonds directly involved in this process [49]. Most likely, the increase of viscosity and SME due to CWC addition are due to the higher solid volume fraction F_v, by analogy with the viscous behavior of a suspension. However, the dependence of K as a function of F_v cannot be satisfactorily described by a usual rheological model of suspension, possibly because of various effects of CWC addition [50]. Finally, the role of mixing is also to entrap air, in order to promote expansion and cellular structure

creation during fermentation, a purpose for which pressure changes provides another degree of

freedom, whilst the rheological properties of degassed dough may be assumed constant [51].

Structural changes during mixing (& methodology)

Rheological properties reflect the dough structure, and rheological models are more reliable and efficient when they are based on structural features. Describing this link is an important research topic for the domain. At the microscopic level, dough can be considered as a composite material, composed roughly of starch grains and aggregated proteins (the gluten network), also containing dietary fibers originating from the starchy endosperm (e.g pentosans in wheat white flour) and possibly from the outer part of the grain (bran) in the case of whole grain flours), mixed with other ingredients (water, fat, sugar, etc.) (Figure 3). Many works have determined the modifications of protein network during mixing through biochemical analyses (see for instance [42, 43, 52]), whilst imaging techniques and non-destructive methods, have been much less employed, although complementary. Ultra sound is a good example of an efficient in-line method that can evaluate dough rheological properties and explore air bubbles size distribution, provided deconvolution between water distribution and gluten network development has been performed [53].

Vibrational spectroscopic techniques have provided valuable insights on dough protein secondary structure [54]. The NIR spectra modifications recorded during mixing have been linked to physical and chemical mechanisms taking place inside the dough [55], whereas amide bands identified by MIR spectroscopy suggested that dough development was linked to the ratio β -sheets / random coil [56]. Conformational changes studied by FT-Raman spectroscopy enabled to put forward that the decrease of dough extensibility due to fiber addition can be attributed to the increase of β -sheets [57]. By applying fluorescence spectroscopy completed with chemometrics, the dough mixing phases (hydration, dough development, and stability and softening) were clearly separated and different wheat flours could be identified on the basis of their bread baking performance [58].

Although partly destructive, the environmental scanning electronic microscopy (ESEM) has shown that dough mixing led to starch granules embedded in a protein matrix of gluten strands (also see Figure 3), coinciding with lower tan δ value (=G"/G') [59]. At a larger scale, confocal scanning laser microscopy (CSLM) with appropriate staining, evidenced different gluten/starch morphologies according to SME reflecting different mixing and deformation mode. The heterogeneity in the form of gluten aggregates appearing at larger SME reflected dough overmixing [43]. CSLM images can be analysed by protein network analysis (PNA), thus, lacunarity (i.e. amount and size of network gaps) was found to be related to the shear viscoelastic modulus G* for dough obtained by mixing with gluten modifying agents [60, 61].

Whatever dough morphology, water distribution among components is an important factor for dough rheological properties. It continuously evolves during mixing and cannot be simply inferred from the water holding capacity of individual component [62]. Low Resolution Proton Nuclear Magnetic Resonance (LR 1H NMR) — and most specifically the Carr-Purcell-Meiboom Gill (CPMG) pulse sequence — has been used to investigate the relationship between biopolymer interactions and water dynamics responsible for the evolution of the rheological properties of dough, flour model systems and bread, since the 1970s [63]. In dough, different proton populations were observed and tentatively assigned to water more or less linked with main flour constituents, i.e. starch and gluten [64]. The evolution of the proton populations has been studied during mixing [65], storage [66, 67], when changing flour/dough composition [68, 69] or according to dough hydration level [70, 71, 65]. It was interpreted as chemical/physical transformations of the flour constituents. For example, the increase of dough hydration level impacts only the most mobile fraction of protons, which corresponds to the free water outside starch granule [64]. Both the amount of protons and their mobility increased with water [71, 65], reflecting a lower rigidity of the dough, as observed by a decrease of modulus G measured by DMA [70]. These recent works can describe the changes of

distribution of water in dough and open prospects for a better understanding of the changes of dough rheological properties at mixing.

So integrating the complementary information brought by these different methods should be an important research perspective to provide rheological measurements and models with sound structural basis.

Modelling dough at mixing

Modelling can help to provide insights on the mechanisms involved in dough changes, spare machine time devoted to experimental trials, simulate process conditions in order to predict their impact on dough and bread properties, allow an accurate control to maintain optimal quality. In the field of materials manufacturing, the use of numerical software is now common practice from the design to the process and logistics control, and part of the Industry 4.0 approach. Likely because of the lack of knowledge about physical properties of foods and the underlying mechanisms of structural changes, it is less common in food processing, although numerical modelling has been developed in food processing specially by applying Computational Fluid Dynamics [72]. This approach, also called deterministic, or mechanistic, mostly relies on the theoretical frame of Continuum Mechanics. It is complex and, besides strong investment, needs to be fueled by a rheological model (Figure 1). In a complementary manner, experimental approaches may be guided by statistical models which require the fitting of experimental data according to numerical procedure and rationale. This approach can lead to the optimization of a product or a process in shorter time; such models are data-driven models [73]. Both approaches – deterministic and data driven, have in common to represent, in various mathematical forms, the relations between input and output variables.

Data-based models

These models have in common to rely on the data directly obtained from experiments on the process itself. They include classical approaches in food science and technology, and more recent ones that have gained considerable interest since the developments of machine learning (artificial intelligence) [74]. Many works have used experimental design and response surface models, for instance to determine the mixing conditions (time, power) that lead to an optimized cellular structure after fermentation [75]. Likewise, baking results were predicted from farinograph measurements using stepwise linear regression and artificial neuronal networks [76]. Artificial neural networks were also used to predict the power delivered to the dough and its temperature for various mixing conditions

[77]. The analysis of power consumption, deduced from torque measurement, or Farinograph consistency, or profiles P(t), provides a simple mean to test various factors such as mixer filling, dough moisture [78], and determine extension properties for different wheat varieties [79]. The redistribution (migration) of water between starch, gluten and fibers in model bread dough has been determined from the changes of dough consistency during mixing [68]. Despite good predictive power, the model validity is generally limited by the dataset used to "learn" the parameters, i.e. the experimental domain. Integrating the physical understanding of the dough behaviour in the models is a way to improve robustness by limiting aberrant prediction out of the domain of validity.

In this view, between data-driven and deterministic models, the concept of basic knowledge model (BKM) has been proposed as a practical medium to transfer and implement food science knowledge beyond the laboratory [80, 81]. Rheological models (e.g. Eq. 1,2) can be considered as BKM, because they are easy to handle and they capture essential knowledge about material behaviour. Actually, no deterministic model, based on continuum mechanics, can describe all physical mechanisms involved during dough mixing, whereas, at industry level, the design of baked products is still relying on experts 'know-how. Knowledge-based models have been proposed to integrate this expertise with scientific understanding of the dough rheology, in order to predict quality criteria from flour characteristics and process conditions [82, 83].

Deterministic (Mechanical) models

Dough mixing is a complex operation because the material is non-Newtonian, viscoelastic and evolutive, hence it has a complex constitutive equation; moreover, the geometry is complicated with a free surface, since the dough surface continuously changes because of the motions of the mixing arm and bowl. Therefore, analytical mechanical models of planetary mixers, based on dimensional analysis and applied to Newtonian fluids, only predict mixer's performances [84] whereas the solution of momentum equations require numerical methods like FEM (Finite Element Modelling), implemented by Computational Fluid Dynamics (CFD). Therefore, dough mixing has first been modelled using a Newtonian model for viscosity in order to determine the stream lines and shear rate fields [85], whereas various non-Newtonian models have been tested in a 2D geometry mixer [86]. Both complexities, geometry and rheological behavior, have been taken into account recently, leading to a satisfactory description of dough flow patterns [87]. In this study, the rheological behaviour was described by the joined use of two models, namely White-Metzner, that involves a single relaxation time, and Bird-Carreau, which is essentially similar to eq. (1). Further, using CFD, the flow of dough, behaving as a power law fluid, in tube and contraction geometries was simulated

to identify the strain domains that cause damage and unstable process regions [88]. Such simulations can be useful not only to optimize mixer geometries and processes conditions but also to capture structural changes of dough. The selection of the rheological model for simulations studies results from a trade-off between the accuracy of the prediction and the costs of computation, but recent progresses in computational resources suggest that more comprehensive rheological models can be integrated in a near future.

Conclusions

This review shows that in spite of the many studies dedicated to dough rheology and mixing, the structure of dough microstructure has not been related to its rheological properties yet, and in turn, to its behaviour during processing. Modelling this operation, either by deterministic or data driven approaches is certainly a determinant support with that regard. Maybe in a near future, "smart" mixers will stop automatically the mixing in anticipation of an exaggerated drop of the dough viscosity, or in case of obvious underhydration, resulting from an error of a water dosage. Besides process control, the sourcing of raw material, and especially wheat flour, could be improved by specifying further dough processing criteria, thanks to a better knowledge of its rheology. Recent advances in spectroscopic methods, especially NMR, open perspectives to improve these aspects. Actually, climate change leads to an evolution of the wheat production worldwide while the demand for healthier and safer food changes the classical view of product quality. These transitions impose a better control of wheat quality, of the process, and, more generally, a better knowledge of the rheological properties of food materials.

Declaration of interest:

Nothing declared

Figure 1: Conceptual view of the relationships between dough structure, rheological properties and mixing process. Continuous arrows mean "influences" whereas dotted ones mean "gives information about". Rheological model is also called "constitutive equation". Please note that rheology under small deformations, i.e. fundamental, can also lead to constitutive models but not applicable to modelling process, because of too different strain conditions.

Figure 2: Examples of time-power (P) curves recorded during dough mixing (m=3kg) for different rotation speeds of the spiral (O: 320 rpm, •: 200 rpm, •: 80 rpm) and energy (dotted line at bottom) for 200rpm, adapted from [44].

Figure 3: Different levels of matter organisation of wheat flour dough during mixing: from left to right: straight from the mixer, CSLM image (width 500 μm) of composite morphology (starch: green, protein: red) with air bubbles, SEM image (width 50 μm) of starch granule entrapped in gluten filaments, arrangement of gluten β—sheets in water environment (from [89]), and protein interactions (dotted and cont.: H₂ and S-S bonds, resp.) from [90]. Images from INRAE.

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References and recommended reading

- 324 Papers of particular interest, published within the period of review, have been highlighted as:
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- Useful and thorough review about the different methods to control dough mixing, especially optimal consistency for various equipments. Processing variables during mixing (e.g. torque) are related to structural aspects of dough, inc. gluten network, through various analytical methods.
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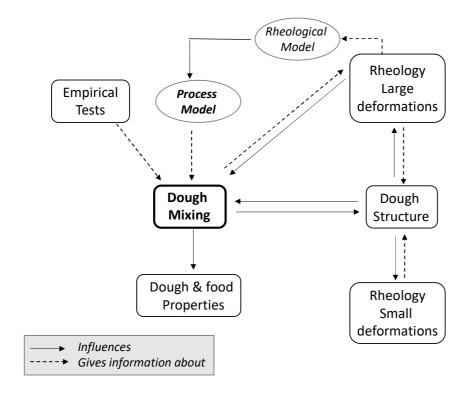


Fig.1 Dough Rheology Mixing Della Valle et al.

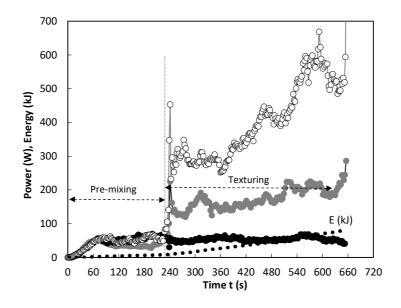


Fig.2 Dough Rheology Mixing Della Valle et al.

