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1	Rheology of wheat flour dough at mixing
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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.
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#### 22 Introduction

23 Anticipating the mechanical behaviour of the wheat flour dough is a major issue for the baking

- 24 industry. With the increasing variability of the wheat quality, due to environmental changes or to
- 25 sourcing diversification, it becomes essential to comprehensively assess the common rheological
- 26 models in a review with a view to examine their capacity to support decision in industry.
- 27 Interestingly, most rheological properties of the dough are determined as soon as the mixing
- 28 operation, the first operation of the processes. The mixing brings the mechanical energy and the
- 29 deformation required to distribute and hydrate homogenously the flour constituents and to form the
- 30 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  $\geq$  0.1) is actually trapped
- during mixing [1], which conditions the fermentation and eventually many quality criteria of bakery
- 34 products. This includes nutritional properties as well, such as Glycaemic Index and dietary fiber
- 35 content [2, 3], also strongly dependent of the dough rheology at mixing.
- 36 The present review offers a concise overview of the advances in dough rheology techniques and
- 37 models and propose guidelines for improving suitability to issues of the baking industry.
- 38

#### 39 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).

43

# 44 Empirical testing

45 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 46 methods, to mimic processing conditions [4]. The Farinograph® measures the torque developed 47 48 during kneading in order to draw a consistency curve which presents a peak indicating the optimum 49 development of the dough; the Extensograph<sup>®</sup> measures the force required to stretch a dough 50 cylinder down its middle to determine its resistance; the Rheofermentometer<sup>®</sup> follows the gas release of a dough under stress, and thus predicts its ability to expand during fermentation; the 51 52 Alveograph<sup>®</sup> 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 53

54 popular among professionals, and still receives scientific attention, e.g. for determining the

- 55 technological value of the wheat varieties [5]. Although these methods may be reproducible when
- 56 conveniently handled, their results, generally expressed in arbitrary units, can only be used to
- 57 perform relative comparison of a set of flours and the test conditions are generally insufficient to
- 58 support a physical interpretation based on the flour composition or the dough structure. Indeed,
- 59 they involve different modes of deformation simultaneously in an uncontrolled way, which prevents
- 60 the relevant determination of intrinsic rheological properties in relation to the dough structure.

# 61 Small deformations ("fundamental rheology")

Dynamic shear measurements are carried out at low strains ( $\leq 0.1\%$  for wheat flour doughs), i.e. in 62 63 the linear viscoelastic domain (LVD). In practice, they require a few grams of dough prepared in small 64 laboratory kneaders, such as the Mixograph<sup>®</sup>, where the continuous torque measurement can detect 65 peak formation, which reflects the optimum consistency of the dough [6]. These testing conditions 66 are far from those encountered in the process, and do not generally lead to good correlations 67 between the results, i.e. dough viscoelastic properties, with flour processing performances [7]. 68 Nevertheless, they have shown that the dough behaves more like a solid than a liquid (G '> G' '), and 69 highlighted the role of high molecular weight glutenins (HMWG) and their entanglements in the 70 creation of the gluten network [8], whilst dough viscoelastic behavior could be represented by a 71 weak gel power law model, the exponent of which reflects the network connectivity [9]. At higher 72 strain, beyond LVD, large amplitude oscillatory shear tests applied to gluten gels, combined with this 73 mathematical model, show that network connectivity decreases and evidence segments extensibility 74 [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 75 76 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].

# 83 Large deformations

Measurements performed in large deformations can be associated to the behavior of the dough
 during processing. The shear viscosity η (γ ) 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  $\eta$  ( $\dot{\gamma}$ ) [12, 17, 18], that can be fitted by the Cross model:

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

90 where  $\eta_0$  is the plateau (Newtonian) viscosity; at higher shear rate, viscosity decreases according to 91 a power law with a slope - n; the transition between the two regimes may be identified by the value 92 of  $\dot{\gamma_0}$  (s<sup>-1</sup>), the reverse of which can be considered as a relaxation time of the dough, of one hour as 93 an order of magnitude.

94 Extension is involved in many food processes and it induces a deformation that implies the 95 deformation of the structural entities (molecules, aggregates, droplets, etc) along the streamlines 96 [19]. The ring stretch test, popularized as Kieffer test, provides a measurement of the uniaxial 97 elongational viscosity [20], while the Extensional Viscosity Fixture is used to characterize the dough 98 strain-hardening behaviour with a better control of strain [21]. The bi-extensional, or biaxial 99 elongation, viscosity,  $\eta_{e}$ , is the property by which the dough resists the growth of gas cells during 100 fermentation, highlighting its strain-hardening behavior, and reflecting its capacity for gas retention 101 and stability of the dough at rest [22].  $\eta_e$  can be determined by a uniaxial compression of the dough 102 between lubricated plates (Lubricated Squeezing Flow, LSF) [23, 24]. In the case of dough made from 103 cereal flours, the measurements of the stress  $\sigma$ , of the equi-biaxial strain  $\epsilon_{e}$ , and of the strain rate  $\dot{\epsilon_{e}}$ , 104 lead to the determination of  $\eta_e$  according to the following equation [25, 26]:

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

105 where K and m are the consistency and flow indices, respectively, defined at constant strain value, 106 SHI being the shear hardening index (> 1). Note that the consistency index K should not be 107 confounded with the consistency derived from torque curves recorded on instrumented mixers 108 (Farinograph, Mixograph, etc). Although dough behaves like a solid rather than a fluid, it never 109 reaches a true state of equilibrium. However, the comparison of the results of LSF tests carried out at 110 constant displacement speed with those obtained at constant strain rate [27], shows that, at 111 constant strain,  $\eta_e$  can be expressed by a power law as deduced from equation (2). Bi-extensional 112 properties can also be determined by the bubble inflation test [28]. The results of this test can be 113 related to those of the Chopin Alveograph [29, 30], provided the accurate measurement of the dough 114 film thickness [31], and  $\varepsilon_e \le 1.5$ , which actually holds for the proofing stage [32]. The same group has 115 determined the shear and extension behaviours, inc. the use of LSF, and proposed a composite

116 micromechanical model that evidenced the decohesion between gluten network and starch grains 117 [33]. Note that mathematical expressions of stress, modulus or viscosity, as function of strain, and 118 strain rate, which are simply illustrated by eq.1-2, are mathematical rheological models, also called 119 constitutive equations (see Figure 1). More accurate mathematical models may be found in the 120 literature, especially to take into account small and large strains, shear and elongational 121 deformations, and time dependence. Finally, rheological tests are mostly performed on unyeasted 122 dough, although fermentation and metabolites have been shown to reduce slightly extensibility and 123 extensional viscosity [34, 35], which was attributed to gluten network degradation [36].

124

#### 125 Dough mixing follow-up

126 Various mixer geometries and working conditions affect bread dough readiness, delineated by 127 adequate mixing time and level of water added [37]. Dough readiness is usually assessed by an 128 optimum consistency target at mixing with the Farinograph, which is a sound and practical indicator 129 for the baker. However, it remains difficult to relate this property with rheological properties, 130 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 131 132 samples induces distinct mechanical treatments which likely clouds the relations between the results 133 [40].

134 The dough development along mixing is followed by the measurement of the power (or torque) 135 curve P(t), and of the Specific Mechanical Energy (SME) deduced from the area under the curve 136 (Figure 2). For wheat, values of SME for an optimum dough development generally stand in the interval [20-60 kJ.kg<sup>-1</sup>] [39, 41]. The negative correlation between the SME and storage ratio 137 138 measured by DMA shows that SME contributes to the creation of the gluten network [41]. This 139 relation is further specified through the decoupling of the mechanical and kinetic effects on the 140 unextractable fraction of glutenin polymers [42]. The optimum SME value depends on the type of 141 deformation mode which results from the mixer geometry. Indeed, with a z-blade and planetary 142 mixers, shear and extensional deformations contribute in a distinct manner to the development of 143 dough structure [41, 43]. By analogy with fluid mixing principles [42, 44], whatever the mixer 144 geometry, the shear viscosity  $\eta$  can be evaluated through power measurements:

145

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

146  $t_m$  being the mixing time and  $\rho_d$  the density of the dough ( $\approx$ 1.2 kg.L<sup>-1</sup> [3]),  $A_m$  is a constant,

147 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<sub>m</sub> (= 1.55 for a z-mixer with a capacity of 5 kg) [41]). A<sub>m</sub>
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].

153 Besides its use for rheometry, P(t) can monitor dough changes, for instance when incorporating 154 ingredients to the flour such as insoluble wheat components (CWC) to increase the dietary fiber 155 content, a huge challenge in baking industry. When CWC are added, SME increases, and so does the 156 dough consistency index (K in Eq. (2)), while the ratio (E'max / E'min) measured by DMA decreases 157 [48]. The increase of the dough consistency index K is unlikely to be the result of a higher gluten 158 network structuring since CWC can inhibit the creation of S-S bonds directly involved in this process 159 [49]. Most likely, the increase of viscosity and SME due to CWC addition are due to the higher solid 160 volume fraction  $F_v$ , by analogy with the viscous behavior of a suspension. However, the dependence 161 of K as a function of  $F_v$  cannot be satisfactorily described by a usual rheological model of suspension, 162 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].

166

#### 167 Structural changes during mixing ( & methodology)

168 Rheological properties reflect the dough structure, and rheological models are more reliable and 169 efficient when they are based on structural features. Describing this link is an important research 170 topic for the domain. At the microscopic level, dough can be considered as a composite material, 171 composed roughly of starch grains and aggregated proteins (the gluten network), also containing 172 dietary fibers originating from the starchy endosperm (e.g pentosans in wheat white flour) and 173 possibly from the outer part of the grain (bran) in the case of whole grain flours), mixed with other 174 ingredients (water, fat, sugar, etc.) (Figure 3). Many works have determined the modifications of 175 protein network during mixing through biochemical analyses (see for instance [42, 43, 52]), whilst 176 imaging techniques and non-destructive methods, have been much less employed, although 177 complementary. Ultra sound is a good example of an efficient in-line method that can evaluate 178 dough rheological properties and explore air bubbles size distribution, provided deconvolution 179 between water distribution and gluten network development has been performed [53].

180 Vibrational spectroscopic techniques have provided valuable insights on dough protein 181 secondary structure [54]. The NIR spectra modifications recorded during mixing have been linked to 182 physical and chemical mechanisms taking place inside the dough [55], whereas amide bands 183 identified by MIR spectroscopy suggested that dough development was linked to the ratio  $\beta$ -sheets / 184 random coil [56]. Conformational changes studied by FT-Raman spectroscopy enabled to put forward 185 that the decrease of dough extensibility due to fiber addition can be attributed to the increase of  $\beta$ -186 sheets [57]. By applying fluorescence spectroscopy completed with chemometrics, the dough mixing 187 phases (hydration, dough development, and stability and softening) were clearly separated and 188 different wheat flours could be identified on the basis of their bread baking performance [58].

189 Although partly destructive, the environmental scanning electronic microscopy (ESEM) has 190 shown that dough mixing led to starch granules embedded in a protein matrix of gluten strands (also 191 see Figure 3), coinciding with lower tan  $\delta$  value (=G"/G") [59]. At a larger scale, confocal scanning 192 laser microscopy (CSLM) with appropriate staining, evidenced different gluten/starch morphologies 193 according to SME reflecting different mixing and deformation mode. The heterogeneity in the form 194 of gluten aggregates appearing at larger SME reflected dough overmixing [43]. CSLM images can be 195 analysed by protein network analysis (PNA), thus, lacunarity (i.e. amount and size of network gaps) 196 was found to be related to the shear viscoelastic modulus G\* for dough obtained by mixing with 197 gluten modifying agents [60, 61].

198 Whatever dough morphology, water distribution among components is an important factor 199 for dough rheological properties. It continuously evolves during mixing and cannot be simply inferred 200 from the water holding capacity of individual component [62]. Low Resolution Proton Nuclear 201 Magnetic Resonance (LR 1H NMR) – and most specifically the Carr-Purcell-Meiboom Gill (CPMG) 202 pulse sequence – has been used to investigate the relationship between biopolymer interactions and 203 water dynamics responsible for the evolution of the rheological properties of dough, flour model 204 systems and bread, since the 1970s [63]. In dough, different proton populations were observed and 205 tentatively assigned to water more or less linked with main flour constituents, i.e. starch and gluten 206 [64]. The evolution of the proton populations has been studied during mixing [65], storage [66, 67], 207 when changing flour/dough composition [68, 69] or according to dough hydration level [70, 71, 65]. 208 It was interpreted as chemical/physical transformations of the flour constituents. For example, the 209 increase of dough hydration level impacts only the most mobile fraction of protons, which 210 corresponds to the free water outside starch granule [64]. Both the amount of protons and their 211 mobility increased with water [71, 65], reflecting a lower rigidity of the dough, as observed by a 212 decrease of modulus G measured by DMA [70]. These recent works can describe the changes of

213 distribution of water in dough and open prospects for a better understanding of the changes of

214 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.

218

## 219 Modelling dough at mixing

220 Modelling can help to provide insights on the mechanisms involved in dough changes, spare 221 machine time devoted to experimental trials, simulate process conditions in order to predict their 222 impact on dough and bread properties, allow an accurate control to maintain optimal quality. In the 223 field of materials manufacturing, the use of numerical software is now common practice from the 224 design to the process and logistics control, and part of the Industry 4.0 approach. Likely because of 225 the lack of knowledge about physical properties of foods and the underlying mechanisms of 226 structural changes, it is less common in food processing, although numerical modelling has been 227 developed in food processing specially by applying Computational Fluid Dynamics [72]. This 228 approach, also called deterministic, or mechanistic, mostly relies on the theoretical frame of 229 Continuum Mechanics. It is complex and, besides strong investment, needs to be fueled by a 230 rheological model (Figure 1). In a complementary manner, experimental approaches may be guided 231 by statistical models which require the fitting of experimental data according to numerical procedure 232 and rationale. This approach can lead to the optimization of a product or a process in shorter time; 233 such models are data-driven models [73]. Both approaches – deterministic and data driven, have in 234 common to represent, in various mathematical forms, the relations between input and output 235 variables.

#### 236 Data-based models

237 These models have in common to rely on the data directly obtained from experiments on the process 238 itself. They include classical approaches in food science and technology, and more recent ones that 239 have gained considerable interest since the developments of machine learning (artificial intelligence) 240 [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 241 242 fermentation [75]. Likewise, baking results were predicted from farinograph measurements using 243 stepwise linear regression and artificial neuronal networks [76]. Artificial neural networks were also 244 used to predict the power delivered to the dough and its temperature for various mixing conditions

8

245 [77]. The analysis of power consumption, deduced from torque measurement, or Farinograph 246 consistency, or profiles P(t), provides a simple mean to test various factors such as mixer filling, 247 dough moisture [78], and determine extension properties for different wheat varieties [79]. The 248 redistribution (migration) of water between starch, gluten and fibers in model bread dough has been 249 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 250 251 experimental domain. Integrating the physical understanding of the dough behaviour in the models 252 is a way to improve robustness by limiting aberrant prediction out of the domain of validity.

253

254 In this view, between data-driven and deterministic models, the concept of basic knowledge model 255 (BKM) has been proposed as a practical medium to transfer and implement food science knowledge 256 beyond the laboratory [80, 81]. Rheological models (e.g. Eq. 1,2) can be considered as BKM, because 257 they are easy to handle and they capture essential knowledge about material behaviour. Actually, no 258 deterministic model, based on continuum mechanics, can describe all physical mechanisms involved 259 during dough mixing, whereas, at industry level, the design of baked products is still relying on 260 experts 'know-how. Knowledge-based models have been proposed to integrate this expertise with 261 scientific understanding of the dough rheology, in order to predict quality criteria from flour 262 characteristics and process conditions [82, 83].

263

#### 264 Deterministic (Mechanical) models

265 Dough mixing is a complex operation because the material is non-Newtonian, viscoelastic and 266 evolutive, hence it has a complex constitutive equation; moreover, the geometry is complicated with 267 a free surface, since the dough surface continuously changes because of the motions of the mixing 268 arm and bowl. Therefore, analytical mechanical models of planetary mixers, based on dimensional 269 analysis and applied to Newtonian fluids, only predict mixer's performances [84] whereas the 270 solution of momentum equations require numerical methods like FEM (Finite Element Modelling), 271 implemented by Computational Fluid Dynamics (CFD). Therefore, dough mixing has first been 272 modelled using a Newtonian model for viscosity in order to determine the stream lines and shear 273 rate fields [85], whereas various non-Newtonian models have been tested in a 2D geometry mixer 274 [86]. Both complexities, geometry and rheological behavior, have been taken into account recently, 275 leading to a satisfactory description of dough flow patterns [87]. In this study, the rheological 276 behaviour was described by the joined use of two models, namely White-Metzner, that involves a 277 single relaxation time, and Bird-Carreau, which is essentially similar to eq. (1). Further, using CFD, 278 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.

- 285
- 286

# 287 Conclusions

288 This review shows that in spite of the many studies dedicated to dough rheology and mixing, the 289 structure of dough microstructure has not been related to its rheological properties yet, and in turn, 290 to its behaviour during processing. Modelling this operation, either by deterministic or data driven 291 approaches is certainly a determinant support with that regard. Maybe in a near future, "smart" 292 mixers will stop automatically the mixing in anticipation of an exaggerated drop of the dough 293 viscosity, or in case of obvious underhydration, resulting from an error of a water dosage. Besides 294 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 295 296 advances in spectroscopic methods, especially NMR, open perspectives to improve these aspects. 297 Actually, climate change leads to an evolution of the wheat production worldwide while the demand 298 for healthier and safer food changes the classical view of product quality. These transitions impose a 299 better control of wheat quality, of the process, and, more generally, a better knowledge of the 300 rheological properties of food materials.

301

## 302 Declaration of interest:

303 Nothing declared

#### 304 List of figures

- 305 Figure 1: Conceptual view of the relationships between dough structure, rheological properties and
- 306 mixing process. Continuous arrows mean "influences" whereas dotted ones mean "gives information
- 307 about". Rheological model is also called "constitutive equation". Please note that rheology under
- 308 small deformations, i.e. fundamental, can also lead to constitutive models but not applicable to
- 309 modelling process, because of too different strain conditions.
- 310
- 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].
- 314
- 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  $\beta$ -sheets in water environment (from [89]), and protein
- interactions (dotted and cont.:  $H_2$  and S-S bonds, resp.) from [90]. Images from INRAE.
- 320

321

322

### 323 References and recommended reading

- 324 Papers of particular interest, published within the period of review, have been highlighted as:
- 325 of special interest
- **326** •• of outstanding interest
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Fig.1

Dough Rheology Mixing

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Fig.2Dough Rheology MixingDella Valle et al.

