|  |  |
| --- | --- |
| cetlogo ***CHEMICAL ENGINEERING TRANSACTIONS***  ***VOL. , 2023*** | A publication of  aidiclogo_grande |
| The Italian Association  of Chemical Engineering  Online at www.cetjournal.it |
| Guest Editors: Laura Piazza, Mauro Moresi, Francesco Donsì  Copyright © 2023, AIDIC Servizi S.r.l. **ISBN** 979-12-81206-01-4; **ISSN** 2283-9216 | |

Non-intrusive experimental monitoring of gluten-free dough mixing in 1L scale mixer using Electrical Resistance Tomography

Federico Alberini \*, Giuseppina Montante, Alessandro Paglianti

Department of Industrial Chemistry “Toso Montanari”, Laboratory of Applied Fluid Dynamics and Mixing - Alma Mater Studiorum – Università di Bologna, via Terracini 34, 40131 Bologna, Italy.

\*federico.alberini@unibo.it

In this study, for the first time Electro Resistance Tomography is employed for macroscale (bulk) characterization of gluten free dough during mixing in 1L scale vessel. Gluten free dough is a complex fluid, which presents a non-Newtonian behaviour. Its rheological behaviour is complex because it is not only shear rate dependent, but also time dependent. The investigated gluten-free dough consists of a mixture of gluten free flours (chickpeas, corn and potato starch), a thickening agent, instantaneous yeast and water. As thickening agent, Xanthan gum is used, which is a polysaccharide with many industrial uses, including food additive. The dough has been prepared in a 3D printed mixer equipped with double rotation shaft lid. Thus a “pizza” hook rotates off-centre (respect to the lid of the vessel) and on the axis of rotation of the hook itself. At the wall of the vessel, the electrodes for the tomography measurements are added in 4 circular planes.

With the current work, a new approach to dynamically monitor the mixing developments is suggested, showing the potentiality of the technique to not intrusively identify inhomogeneity in the dough during the process. A standard operating procedure is used for the preparation of the dough, which consists in precise steps in time of material addition into the mixture.

1. **Introduction**

Mixing is a critical step in any process, included the preparation of a dough. Generally, this process consists in flour and water forming a dough by both blending and distributing the dough ingredients and developing a certain structure. The energy input contributes to a uniform distribution of the different ingredients (flour, water, yeast, etc.), to the hydration of flour particles (starch, proteins, non-starch polysaccharides, etc.) (Gomez et al., 2013) The dough must be mixed for a specific time, known as optimum dough development, to ensure optimal loaf volume and to ensure a correct final texture for the final products. Stopping before the optimal point results in an under-mixed dough that gives bread of inferior volume and crumb quality. Mixing beyond the optimal development point induces dough stickiness, decreases dough consistency and negatively affects bread quality. In the last few decades, the manufacture of this category of products with the addition of gluten-free flour became more and more relevant. Gluten-free baked products have generally poor quality in terms of volume, texture, and shelf life. As a result, manufacturing high-quality gluten-free baked products has become one of the most difficult challenges for manufacturers, cereal technologists, and scientists (Yazar and Demirkesen, 2022). In the literature, there are several attempts of characterizing the transient behaviour of standard bread dough using techniques such as NIR (Near Infrared Spectroscopy) or MIR (Mid Infrared Spectroscopy) or NMR (Nuclear Magnetic Resonance) (Fanari et al., 2021, Dufour et al., 2023). The MIR and the NIR allow information concerning chemical content and composition of food products to be obtained (Kaddour et al., 2008). Successful results have been presented in several works; however, a clear limitation of the techniques is that they provide very localized information, but mixing of these fluids is far to be homogeneous and industry requires sensor technology which can enable real time monitoring of reacting and not reacting systems (Alberini et al., 2021; Patel et al., 2013).

* 1. Material and methods

The experimental rig consists of a 3D printed mixer (diameter, T=0.065 m, height, H=0.15 m) equipped with double rotation shaft lid as shown in Figure 1a-b. Ad hoc gears have been manufactured (Figure 1a) to provide such double rotation effect providing an off-center rotation to the hook (dh=0.022 m) (Figure 1b), which also rotates on its own axis. For the investigated mixing condition, the hook rotates on its axis at 156 rpm while rotating off-center at 50 rpm. This double rotation has been reproduced to mimic a standard planetary mixer.

|  |  |  |
| --- | --- | --- |
| a | b  **shaft** | c |
| A picture containing floor, indoor  Description automatically generated | **hook** |  |

*Figure 1: a-3D printed parts of the mixer, b-assembled double rotating shaft lid, c- schematic of ERT measurement planes and location of the ingredients feed.*

ERT technique was used to evaluate the mixing performances based on the variation of the mixture conductivity. The conductivity data were collected by the ERT instrument P2000 and recorded and analyzed with the software p2+ V9, both provided from Industrial Tomography Systems Ltd., UK. The measurements were performed adopting the circular adjacent strategy, injecting the current by an adjacent electrodes pair at a time and measuring the voltage difference from the remaining pairs of electrodes. In present work, electric current of amplitude equal to 15 mA and frequency of 9600 Hz were used. Four circular planes equipped with 16 electrodes (Figure 1c) were located equally spaced by 0.015 m. P1 was located 0.015 m from the bottom of the vessel to ensure that with the amount of water initially fed for the dough preparation, the liquid level reached approximately half distance between P1 and P2. This ensured a continuous reliable measurement for P1. The reconstruction of the conductivity map by means of the linearized back projection algorithm was achieved in real time, thus allowing for on-line monitoring. The tomogram was reconstructed using a mesh having cell size of 3.25×3.25 mm, which renders a total of 316 values. Thus, when considering the 4 investigated planes, a total number of 1264 values was obtained. For details about the reconstruction algorithm and about the ERT system please refer to Maluta et al. (2020). The measured variable is the dimensionless conductivity calculated as the conductivity of the mixture in the vessel divided by the reference conductivity. The reference was taken from an average of 100 tomograms with the hook rotating at same speed used for the dough mixing, and the tank filled with 350 mL of water with a concentration of 2 g L-1 of NaCl. The data rate was 0.45 frames per second, corresponding to a temporal resolution of 2.24 s. A master batch of gluten-free-flour mixture (GFFM) was prepared mixing extensively the chickpeas (CF), corn, potato starch flours with the thickening agent, Xanthan gum. To be noticed that both corn and potato starch flours contain residual of salt. This was tested a priori dissolving such flours in deionized water and measuring the mixture conductivity by means of a standard conductivity meter (PC70 Vio). The dough preparation procedure consists of 8 steps namely additions (A1,A2, etc) which are fed through the ingredients feed located on the wall of the tank (Figure 1c). Firstly, 136 g of water, having the same concentration of sodium chloride used for the reference, was added in the mixer. Then 2.45 g of dried yeast is added to the mixture and at this point the ERT recording is started. After 195 s from the addition of the dried yeast, two consecutive additions of 20 g of GFFM are introduced into the system with a time interval between them of approximately 180 s. These are followed by other four additions, keeping the same time interval among them, but with an amount of flour increased to 30 g per addition. After other 180 s from the last addition of GFFM, 3 g of oil is added and finally after other 180 s, the last 5 g of extra chickpeas flour is added. To be noticed, chickpeas flour alone did not increase the conductivity of a water solution as corn and starch flours did. All of this sum up to a total time of circa 1675 s, subsequently the mixture is mixed for other 565 s to allow the system to reach a certain level of homogeneity across the four measured planes. Moreover, to understand visually what was happening at the surface of the dough during the blending, an endoscope (YINAMA 1080P HD) was used to acquire images through the ingredients feed.

* 1. Results and discussion
     1. Dough mixing tracking

The bottom measuring plane, P1, is used to continuously monitor the mixing process given that, only on that plane, the minimum height of the liquid is assured for the whole process. In Figure 2a, an example of a typical tomogram is shown. To track the dough mixing dynamics, the average value of the non-dimensional conductivity is evaluated from the 316 local values. As it can be seen in Figure 2b for the first 85 tomograms (which sums up to 195 s), the normalized conductivity (NC) is constantly oscillating around 0.9, local peaks of the conductivity might be related to the dissolution process of the yeast. The lower value respect to the reference is justified by the addition of yeast which reduces the conductivity compared to the water and salt solution only. Furthermore, in Figure 2b, the additions of flour and other ingredients are highlighted by the yellow dotted lines and red arrows, as the starting point of each addition, then green dotted lines indicate the addition end.

|  |  |
| --- | --- |
| a | b |
|  |  |

*Figure 2: a- example of P1 tomogram (#246, corresponding to time 500s, A3) which consists of 316 cells from which the average is calculated, the colour bar refers to non-dimensional conductivity [-]; b-Plot of the average non-dimensional conductivity for P1 for the 1000 acquired tomograms,*

As it is shown in Figure 2b, A1-6 are the additions of flour, all additions show a similar trend where there is a first decrease (induced by the entrapment of air with the flour) and then an exponential rapid increase of the average conductivity once the flour is wetted. At later stage, the overall system tends to converge to a new conductivity value. Other interesting trend is given by observing the differences between the 20 g (A1-2) versus 30 g (A3-6) additions. From the plot of the conductivity, it seems that the ERT is capable to distinguish the two types of addition (20 g vs 30 g); in fact, the low peaks of conductivity (after each addition of flour, for reference yellow dotted lines) for the 30 g (A3-6) additions decrease their intensities compared to the 20 g (A1-2). This might be related to higher amount of air entrapped in the system. Vice versa, when the flour is wetted, the high peaks of conductivity increase their intensity given more salt is dissolved for the 30 g additions Additionally, other two interesting trends arise by the ERT measurements for the last two additions. As anticipated the A7 is the 3 g of seed oil added to the mixture, here clearly the trend is opposite to the other additions and the average non-dimensional conductivity tends to decrease immediately, as expected given that the oil is a non-conductive fluid. Finally, A8 is the addition of 5 g of chickpeas flour, and in this case the conductivity tends to decrease in the proximity of the addition, given to the non-conductive nature of this flour compared to the mixture. Then, after a long mixing time, the system tends towards a new final conductivity value.

* + 1. Identification of undesired inhomogeneities during the dough mixing process

During a mixing process accumulation of unmixed material in certain parts of the vessel is common, in the case of dough mixing this is even more probable, given the nature of the mixture. As it is shown in Figure 3a, the ERT technique offers a great capability of inhomogeneity detection, in this case due to the flour addition. Both ERT acquisitions and endoscope observations confirm an accumulation of flour near the location of the 13,14,15 electrodes which correspond roughly to the radial location of the ingredient feed. The justification of lower conductivity (~0.7) is due to the presence of unwetted flour entrapping air, which is not conductive.

|  |  |
| --- | --- |
| a | b |
|  |  |

*Figure 3: a-snapshot of #246 tomogram for P*2, b-snapshot acquired with endoscope at same time located above P4.

To confirm the presence of unwanted accumulation of unwetted flour, another example is presented in Figure 4a. This tomogram corresponds to the final tomogram, measured 2240 s after the addition of the dried yeast, which showed a consistent inhomogeneity, again closer to the feed location. The picture, shown in Figure 4b, was taken, after the mixer was turned off and after the removal of the hook that was in front of the electrodes 14,15 and 16. The snapshot from the endoscope showed clearly how the information provided by the ERT measurement were coherent with what happened in the system. More precisely some unwetted flour remained close to the feed nozzle while in the rest of the vessel, where higher value of conductivity was detected, the dough was much more wetted.

|  |  |
| --- | --- |
| a | b |
|  |  |

*Figure 4: a-snapshot of #999 tomogram for P*3, b-snapshot acquired with endoscope at end of the mixing process and removing the dough from the wall where the electrodes 14,15,16 are located.

* + 1. Measurement validation against dynamic filling level

Other interesting information that can be deducted from the analysis of the data collected with the ERT are presented in Figure 5. More in details, in Figure 5a, the number of the values of the non-dimensional conductivity above a nominal value of 3 are reported for the 1000 recorded tomograms. It is worthwhile noticing that a value of the local dimensionless conductivity greater than 3 has no physical meaning, but it is due to a mistake in the image reconstruction due to electrical insolation of some electrodes (Paglianti et al., 2020). This nominal threshold has been selected from the maximum local value reached in P1, in which the liquid level avoids the identification of artefacts of the reconstruction. As it can be seen for P1 and P2, as expected, the number of pixels overpassing this value is practically always equal to zero, however for P3 and P4 this is not the case. The value of the pixels above the threshold drops to zero (#>900) at the end of the experiment when on P3 and P4 no gaps of air or other non-conductive materials are present. To clearly quantify the mixture homogenization, in Figure 5 b the coefficient of variation (CoV) for P4 for # greater than 900 is reported. The CoV is calculated as suggested by Maluta et al. (2020). When the out-of-range values (identified in the presented tomograms of Figure 5b with red pixels) are identified in the tomograms, the CoV results in high values. Once such out-of-range values decrease, the CoV drop to lower value (around tomogram #930), and at this point on the measuring plane all the electrodes work properly, and the measurement can be considered physically acceptable. After the tomogram #930, it is also clear how the CoV value is tending to a lower value with a moderate slope which is due to the long mixing time required to the system to reach a complete homogeneity. This is a powerful feature that ERT offers, enabling the possibility to control the degree of homogenization of the mixture.

|  |  |
| --- | --- |
| a | b |
|  |  |
| c | d |
|  |  |

*Figure 5: a- Number of out-of-range values per tomogram for each plane of reference (P1-4); b- CoV and number of out of range values for P4 between #900 and #999 tomograms; c-* snapshot acquired with endoscope for the tomogram #900, showing the presence of air gap at the wall; d- snapshot acquired with endoscope for the tomogram #950, showing air gap is disappeared at the wall.

Finally, in Figure 5 c and d, two images taken with the endoscope, are presented. Figure 5c is acquired around the tomogram #900, the picture shows the presence of a large air gap close the vessel wall. This presence was also identified by the ERT that was not able to correctly reconstruct the image, see the presence of large zones depicted in red in the Figure 5b. Once the mixing further evolves, tomogram #950 (Figure 5d), the air gap close to the vessel wall disappears, as correctly identified by ERT, indeed the relative tomogram shown in Figure 5b does not present anymore out of range values (pixel depicted in red).

* 1. Conclusions

With this work we presented, for the first time, how ERT measurements can be a rich source of information to track even a difficult process as the mixing of a gluten free dough. An experimental rig developed using 3D printing technology is presented, which offers the possibility of mixing and measuring non-intrusively the mixing dynamics of dough. The mixing phenomena are clearly identified by the non-intrusive measurement, tracking several different additions of ingredients in the mixing system. Moreover, the presence of inhomogeneities, which can affect the quality of the final dough, can be identify by the ERT measurement and the filling level, which the system reached during the process.

Nomenclature

A – Addiction

CF – chickpeas flour

CoV – Coefficient of Variation, -

dh–hook diameter, m

ERT – Electro Resistance Tomography

GFFM – gluten-free-flour mixture

H – total height of the tank, m

NC, – Normalized conductivity, -

Rpm – Revolution per minute, rpm

P – ERT plane

T – Tank diameter, m

-

Acknowledgments

We would like to acknowledge the contribution of Mr Aurelio Pio Di Matteo who supported part of the experimental campaign of this work.

References

Aït Kaddour, A., Mondet, M., & Cuq, B. (2008). Application of two-dimensional cross-correlation spectroscopy to analyse infrared (MIR and NIR) spectra recorded during bread dough mixing. Journal of Cereal Science, 48(3), 678–685. <https://doi.org/10.1016/J.JCS.2008.03.001>

Alberini, F., Bezchi, D., Mannino, I. C., Paglianti, A., & Montante, G. (2021). Towards real time monitoring of reacting species and pH coupling electrical resistance tomography and machine learning methodologies. Chemical Engineering Research and Design, 168, 369–382. <https://doi.org/10.1016/J.CHERD.2021.02.024>

Dufour, M., Foucat, L., Hugon, F., Dugué, A., Chiron, H., Della Valle, G., Kansou, K., & Saulnier, L. (2023). Water mobility and microstructure of gluten network during dough mixing using TD NMR. Food Chemistry, 409, 135329. <https://doi.org/10.1016/J.FOODCHEM.2022.135329>

Fanari F., Keller J., Desogus F., Grosso M., Wilhelm M. (2021). Impact of water and flour components in dough investigated through low-field nuclear magnetic resonance. Chemical Engineering Transactions, 87, pp. 289 – 294. DOI: 10.3303/CET2187049

Gómez, M., Talegón, M., de la Hera, E. Influence of Mixing on Quality of Gluten-Free Bread 2013 Journal of Food Quality 36(2), pp. 139-145

Maluta, F., Montante, G., Paglianti, A., 2020. Analysis of immiscible liquid-liquid mixing in stirred tanks by Electrical Resistance Tomography. Chemical Engineering Science227, 115898. <https://doi.org/10.1016/j.ces.2020.115898>

Paglianti, A., Marotta, G., Montante, G. 2020 Applicability of electrical resistance tomography to the analysis of fluid distribution in haemodialysis modules Canadian Journal of Chemical Engineering 98(9), pp. 1962-1972

Patel, D., Ein-Mozaffari, F., & Mehrvar, M. (2013). Using tomography technique to characterize the continuous-flow mixing of non-newtonian fluids in stirred vessels. Chemical Engineering Transactions, 32, 1465–1470. <https://doi.org/10.33032/CET1332245>

Sinelli, N., Casiraghi, E., & Downey, G. (2008). Studies on Proofing of Yeasted Bread Dough Using Near- and Mid-Infrared Spectroscopy. Journal of Agricultural and Food Chemistry, 56(3), 922–931. <https://doi.org/10.1021/JF0727138>

Ulrici, A., Vigni, M. L., Durante, C., Foca, G., Belloni, P., Brettagna, B., de Marco, T., & Cocchi, M. (2008). At-line monitoring of the leavening process in industrial bread making by near infrared spectroscopy. Journal of Near Infrared Spectroscopy, 16(3), 223–231. https://doi.org/10.1255/JNIRS.781

Yazar, G., & Demirkesen, I. (2022). Linear and Non-Linear Rheological Properties of Gluten-Free Dough Systems Probed by Fundamental Methods. Food Engineering Reviews 2022 15:1, 15(1), 56–85. <https://doi.org/10.1007/S12393-022-09321-3>