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Leavened Dough Processing by Supercritical Fluid Extrusion (SCFX)

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Yeast-leavened dough processing is semicontinuous due to the requirement for fermentation at constant temperature and humidity. Also, new regulations on the emission of alcohols are becoming burdensome on the baking industry. Extrusion processing of dough with supercritical carbon dioxide (SC-CO₂) is envisioned to alleviate emission problems and to decrease production time by eliminating fermentation. A bread dough formulation with 50% (w/w) moisture was leavened by injecting 1.5% (w/w) SC-CO₂ in a twin-screw extruder at 37 °C. Specific mechanical energy input was 260 kJ/kg. The operating apparent shear rate range was 60–260 s⁻¹. SCFX-leavened dough density (420–430 kg/m³) was in good agreement with values reported for similar doughs. The flow behavior index, obtained by an on-line slit rheometer, was 0.49 for the nonleavened control and 0.63 for the SCFX-leavened dough. Apparent viscosity of the SCFX-leavened dough varied from 37 to 23 Pa-s. This new continuous process offers attractive possibilities for industrial applications if further developed.

KEYWORDS: Bread dough; dough density; dough rheology; SCFX; dough additives

INTRODUCTION

Although bread-making is an ancient technology, demand for consistent texture and longer shelf life continues to dominate the narrow profit margins, and thus process control and improvement are constantly sought. The most common industrial bread-making processes are the sponge dough and straight dough processes that need 10-20 min of mixing, 2-3 h of fermentation, and 10-15 min of proofing and loaf forming, followed by 45-60 min of final proofing for a total of 4-5 h (1). Do-Maker and AMFLOW processes are modifications of the sponge dough process that involve prefermentation (2-4 h) followed by the addition of dry ingredients, intense mixing, forming, and final proofing, leading to a decrease in processing time. These processes are used in the United States (1). In CBP, widely used in the United Kingdom, intense mixing (40 kJ/kg) and fermentation take place together (2, 3), leading to a 60% decrease in processing time.

The need for long fermentation and proofing times causes doughmaking to be a semicontinuous operation. Requirement of yeast activity for adequate dough expansion and flavor production have necessitated the development of yeast cultures that retain high activity upon storage and/or under frozen conditions. Such cultures are supplied in dried form and are easily activated (4). Another bottleneck in dough processing is keeping the distribution of gas bubbles even during proofing when the dough gets saturated with carbon dioxide and bubbles tend to coalescence, causing defects in bread (5). Thus, the two opposing choices during final proofing are between maximizing loaf volume and minimizing the number of defects (6).

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The U.S. Environmental Protection Agency (EPA) requirement for lower ethanol emission since 1990 has been another challenge for the bread-making industry. Yeast produces 0.8 kg of alcohol/100 kg of flour, only 0.3% of which is retained and the rest is emitted to the atmosphere. There are ~2500 bakeries in the United States with potential Clean Air Act (CAA) compliance issues due to volatile organic compound (VOC) emissions. Sources located near population centers and emitting >10-25 tons of VOC annually are required to control their emissions. This would include most bakeries because they are located in or near population centers (7). Catalytic oxidation of VOC into CO₂ requires additional investment, which brings about a financial burden on bakeries.

One of the targets of process control in a conventional bakery is adequate but rapid dough development. This, in turn, has necessitated measurement of basic rheological properties. Empirical tests (farinograph, mixograph, alveograph, extensograph, and viscoamilograph) (8) and/or fundamental tests (creep, compression, tension, and flow behavior) (9-14) are used for the rheological characterization of dough. Flow behavior studies on bread dough have been carried out by extruding dough, prepared to 500 Brabender units (BU) in a farinograph, through a rheometer (9-13). Very limited information on the rheology of leavened dough is available in the literature.

A novel technology, termed supercritical fluid extrusion (SCFX) (15-17) is known to impart uniform bubbles (18) by allowing the injection of supercritical carbon dioxide (SC-CO₂) in the extrudate at low temperatures. The high-pressure solubilized SC-CO₂ diffuses out at the extruder die and assumes the role of an expansion agent upon exit (15). The SCFX process permits continuous production of ready-to-bake leavened dough



Figure 1. Typical screw configuration and its corresponding pressure profile developed along the extruder barrel.

where yeast is substituted by $SC-CO_2$ as the leavening agent. As a result, the processing time needed to obtain leavened dough decreases. It also allows an alcohol-free baking environment and good retention of added flavors (19). Additionally, a uniform bubble size distribution achieved by $SC-CO_2$ in leavened dough is expected to minimize dough defects. Incorporation of air does not assume a key role, because nucleation, diffusion, and expansion of dissolved $SC-CO_2$ to the gaseous state overcomes the relative inability of gaseous CO_2 to form bubbles.

Continuous dough processing using SCFX also allows online rheological characterization of nonleavened control and SCFX-leavened dough with a constant processing history, making the comparison of the fundamental rheologies of leavened and nonleavened doughs possible. The objectives of this study were to obtain SCFX-leavened dough with density comparable to the density of conventional yeast-leavened dough and compare their rheological properties. The effect of SC-CO₂ leavening on the rheological properties of nonleavened control dough was assessed at different shear rates while the processing history was kept constant. This is a first attempt to measure dough rheology on-line using a continuous process.

MATERIALS AND METHODS

Ingredients. The dry ingredient formulation was as follows: 100 kg of high-spring dominator flour (ADM Milling Co., Overland Park, KS), 6 kg of sugar, 3 kg of whey protein concentrate (WPC-34) (Custom Ingredients Group, Lomira, WI), 1.5 kg of salt, 0.25 kg of diacetyl-tartaric acid esters of mono- and diglycerides (DATEM) (American Ingredients Co., Kansas City, MO), 0.25 kg of locust bean gum, 0.25 kg of ascorbic acid. Dry ingredients were mixed and fed to the bunker of the extruder. The feed rate of the dry mix was 9.7×10^{-3} kg/s. Melted bakery fat and water were added at the rates of 2.43×10^{-4} kg/s (2.5%, w/w) and 4.85×10^{-3} kg/s (50%, w/w), respectively. For leavening, SC-CO₂ was injected at a rate of 1.46×10^{-4} kg/s (1.5%, w/w).

Extrusion. Bread dough was extruded through the slit die (length $= 2.8 \times 10^{-2}$ m, slit width $= 2.58 \times 10^{-2}$ m, slit height $= 1.6 \times 10^{-3}$ m) of a self-wiping, corotating twin-screw Wenger TX-52 Magnum extruder (Wenger Manufacturing, Sabetha, KS) with a length-to-diameter (L/D) ratio of 28.5:1 utilizing 4.5 heads, and equipped with SC-CO₂ injection ports (**Figure 1**) located at L/D = 24. The product temperature was maintained between 36 and 38 °C. Dough was extruded in the pressure range of 14–15 MPa. The pressure range during rheological characterization varied between 10 and 11 MPa due to the



Figure 2. Schematic of (a) experimental setup and (b) slit-die specifications.

pressure limitations imposed by the slit rheometer. Specific mechanical energy (SME) input to dough development was obtained from the equation

$$SME = (37.3) \times \left(\frac{\% \text{ extruder load}}{100}\right) \times \left(\frac{\text{extruder screw speed}}{306}\right) \times \left(\frac{3600}{\text{extruder feed rate}}\right) (1)$$

where extruder screw speed is in rpm, 306 rpm is the maximum extruder screw speed, 37.3 kW is the power input, and the extruder feed rate is in kg/h. Average SME input was calculated as 260 kJ/kg. Density was measured by weighing dough samples collected in a known volume container. Density results reported were obtained from five extrusion runs performed at a pressure range of 14-15 MPa. Five replicate density measurements were taken both for nonleavened control and for SCFX-leavened dough during each run, and the mean value was reported. Coefficient of variation was 3-5%. Moisture content was measured according to AACC Method 44-19 (20).

The slit rheometer used for rheological measurements was 0.325 m long and had a slit width and height of 5.08×10^{-2} and 3.15×10^{-3} m, respectively (**Figure 2**). The width-to-height ratio was 16, which is generally assumed to be large enough for wall slip effects to be neglected, and the slit length-to-height ratio was 103.2, which is

large enough to assume fully developed flow (21). The slit die had three ports along its length at distances of 0.027, 0.170, and 0.300 m from the die entrance. Calibrated pressure transducers were inserted at the second and third ports to record pressure drop every 10 s (**Figure** 2). A bypass valve was attached to the first port (**Figure 2**) to allow different flow rates through the die and therefore different shear rates at a constant dough history. Mass flow rates through the die and the bypass were measured by weighing material throughput during a 30 s time interval at steady extrusion conditions. Average mass flow rates of 6.7×10^{-3} , 8.3×10^{-3} , 11.1×10^{-3} , 14.0×10^{-3} and 16.8×10^{-3} kg/s were obtained. Simultaneously, corresponding pressure drop data from the pressure transducers were recorded automatically. Data were collected in triplicate. Data reported are the results of four extrusion runs each for nonleavened control and SCFX-leavened dough.

Data Evaluation. Shear stress and shear rate at the wall were calculated from the pressure drop versus mass flow rate data. Wall shear stress (τ_{w} , Pa) was obtained from the equation

$$\tau_{\rm w} = \frac{H}{2(\Delta L)}(\Delta P) \tag{2}$$

where ΔP (Pa) is the pressure drop measured between the two transducers (**Figure 2**), *H* is the slit height (m), and ΔL (m) is the

distance between the two pressure transducers (Figure 2b). Apparent shear rate ($\dot{\gamma}_{app}$, s⁻¹) was obtained from the equation

$$\dot{\gamma}_{app} = \frac{6}{WH^2} \frac{\dot{m}}{\rho} \tag{3}$$

where \dot{m} (kg/s) is the mass flow rate through the slit die, ρ (kg/m³) is the dough density, and W (m) and H (m) are the slit width and height, respectively (**Figure 2**). The error for each calculated shear stress—shear rate combination is between ± 5 and 10% for three replicate measurements at a given pressure drop—mass flow rate combination.

The rheological parameters were evaluated from linear regression of the log-log plot of shear stress (τ_w , Pa) versus shear rate ($\dot{\gamma}_{app}$, s⁻¹) using the power law model given as

$$\tau_{\rm w} = K'(\dot{\gamma}_{\rm app})^n \tag{4}$$

where K' (Pa-s^{*n*}) is the consistency coefficient and *n* (dimensionless) is the flow behavior index. Then, the wall shear rate ($\dot{\gamma}_{w}$, s⁻¹) was calculated using *n* as follows:

$$\dot{\gamma}_{\rm w} = \frac{3n+1}{4n} \dot{\gamma}_{\rm app} \tag{5}$$

Then, apparent viscosity (η_{app} , Pa-s) was calculated as a function of the wall shear rate ($\dot{\gamma}_w$, s⁻¹) from the equation

$$\eta_{\rm app} = K(\dot{\gamma}_{\rm w})^{n-1} \tag{6}$$

where the consistency index, K (Pa-sⁿ), is

$$K = K' \left(\frac{4n}{3n+1}\right)^n \tag{7}$$

RESULTS AND DISCUSSION

Dough Formulation and Processing Conditions. The dough formulation used was similar to that of the white bread recipe in the United States (22) except for relatively higher moisture and ascorbic acid contents and additional ingredients such as bakery fat, locust bean gum, and xanthan gum. SCFX-leavened dough does not contain an active leavening agent; thus, ingredients that promote water absorption and macromolecular modifications are crucial to strengthen the dough structure so as to prevent CO₂ loss following its nucleation and growth at the extruder exit. A higher moisture content was necessary to promote dissolution of SC-CO2. Ascorbic acid was used to enhance the formation of disulfide bonds (23, 24). Locust bean gum and xanthan gum were used to increase water absorption (25, 26). Bakery fat was used to enhance the gas-holding capacity by stabilizing the membrane around bubble nuclei (23). Bakery fat also provides lubrication (23) and protects the gluten-starch network from adverse effects of shear and friction.

The dough formulation was processed in the SCFX machine in which dry ingredients are mixed with the fat phase and hydrated by moisture between L/D = 3 and L/D = 9 (Figure 1). Mixing and conveying along the extruder screw are provided between L/D = 9 and L/D = 23 that result in macromolecular modifications leading to the formation of a network structure with high spring (Figure 1). The likely modifications include formation of the gluten-starch matrix, formation of disulfide bonds, and formation of starch-protein, starch-lipid, and protein-lipid complexes that strengthen the matrix structure (23). SC-CO₂ is injected into the dough matrix at L/D = 24(Figure 1). Reverse flights, located at L/D = 23, bring the dough to the pressure required for SC-CO₂ injection such that the two phases are then mixed and conveyed further along the extruder screw. In zones 3-5 (Figure 1), cooling is provided to keep the material temperature below 40 °C. This is to ensure

 Table 1. Comparison of Dough Densities Obtained by Conventional and SCFX Processing

type of dough	temp (°C)	processing time (s)	mixing pressure (MPa)	density (kg/m ³)
nonleavened control SCFX-leavened nonleavened (literature) ^a	36–38 36–38 30 27	240–300 240–300 180 1400	14–15 14–15 0.1	1080–1100 420–430 1150–1160 700
leavened (literature) ^b	33 37 43	1400 1380 1000		552 420 450

^a Density measured after mixing (28). ^b Density measured after proofing (28).

incorporation of SC-CO₂ into the dough matrix and also to prevent protein denaturation. The restrictor plate at the end of the extruder barrel provides control of the pressure profile (**Figure 1**). The dough is extruded through a slit die that aligns sheared macromolecules imitating the shear and elongation action during conventional bread dough mixing (11). At the die exit, leavening action is accomplished due to a drop in pressure to the atmospheric conditions that cause injected SC-CO₂ to nucleate and grow into bubbles.

Product temperature at the die exit was kept at 36–38 °C, which is within the proofing temperature range for conventional yeast-leavened dough. The SME input (260 kJ/kg) was higher than supplied during dough development in conventional (30 kJ/kg) and CBP (40 kJ/kg) operations (27). This is because of higher pressure development and the pressure profile within the extruder barrel (**Figure 1**) when compared to constant pressure mixing in conventional dough processing.

Dough density was considered as the measure of dough expansion. The density of SCFX-leavened dough (420-430 kg/ m³) was similar to that of the conventionally leavened dough (420 kg/m³) (28, 29) obtained after proofing at 37 °C (Table 1). This shows that $SC-CO_2$ can substitute for the leavening action of yeast under the processing conditions and ingredient formulation described. Also, the use of SCFX technology in leavened dough processing led to a substantial decrease in processing time. It takes only 4-5 min to obtain the ready-tobake leavened dough as compared to 3-5 h in modified versions of the sponge process and 1.5-2 h in CBP (Figure 3). However, the present study is a first attempt at continuous production of leavened dough, and the importance of additional baking studies in analyzing the bread-making potential of SCFX-leavened dough cannot be denied. Although nonleavened and leavened dough densities are the most basic physical properties of dough in quantifying the effect of dough conditioners and improvers and are extensively considered to be essential quality control points in the bread-making industry (28, 29), baking studies are important to implement the overall quality of SCFX-leavened dough on the road to the finished product, the bread.

Dough Rheology. The present study is the first attempt of on-line rheological characterization of leavened dough starting from the dry mix and ending in a nonleavened control or SCFXleavened dough. An experimental arrangement that allows the mass flow rate at different bypass valve positions (**Figure 2**) to be varied enabled evaluation of dough rheology at constant temperature, pressure, and SME input without changing the dough history.

The usual practice is to base the rheological properties of leavened dough on the rheological properties of nonleavened dough (8), which may be fallacious due to decreasing pH, emerging gas bubbles, and differing dough history due to



Figure 3. Comparison of conventional bread dough processing with SCFX technology.

mixing, forming, and proofing. Another approach involves measurement of dough expansion with time during fermentation using a rheofermentometer. The relative relationship between ingredient formulations and the viscosity and elasticity of dough can then be predicted qualitatively using mechanical models (*30*). However, it has not been possible to perform fundamental rheological tests on leavened dough due to the fact that all such techniques that make use of shear, elongation, or compression would disturb the gas bubble distribution and/or lead to the release of CO₂. The absolute effect of leavening on dough rheology is thus not well-known. It is possible to perform online characterization of leavened dough by the application of SCFX technology, because rheological data with constant history can be collected during the production of SCFX-leavened dough as it is extruded.

Regression results (**Table 2**) of the shear stress versus shear rate data (**Figure 4a**) with flow behavior indices of 0.49 for nonleavened control and 0.63 for SCFX-leavened dough characterize the classical shear-thinning behavior of dough (*12*). Dough is a viscoelastic material in which viscosity is combined with considerable elasticity. The power law index is a measure of the resistance of dough against shear rate. Thus, the flow behavior indices obtained for nonleavened control and SCFX-



Figure 4. Rheological behavior of nonleavened control and SCFXleavened dough: (a) flow curves; (b) viscosity curves.

leavened dough indicate that the strength of the dough processed by SCFX technology is comparable with that of a conventional dough. A higher flow behavior index obtained for SCFXleavened dough in comparison with its nonleavened control shows that SC-CO₂ injection renders bread dough less vulnerable to increasing shear rates. This is because the gas phase present in SCFX-leavened dough deforms more easily and acts like an energy absorber, compensating for the shear-thinning effect as compared to the nonleavened control dough.

The consistency coefficients in previous studies were obtained at different dough moisture contents and temperatures (9, 10, 12) (**Table 2**). Thus, the results reported in the literature fall in a wide range ($405-3115 \text{ Pa-s}^n$). Also, within individual studies, the consistency coefficient has been found to vary with dough composition (13) and dough preparation parameters such as mixing time and resting time (10, 13). Also, the data reported show a decreasing trend in consistency coefficient with increasing temperature (10, 12), as expected.

 Table 2. Comparison of On-Line-Measured Rheological Parameters with Previous Studies Involving Off-Line Preparation of Dough Followed by

 Extrusion through a Rheometer

type of mixture	moisture (kg/kg)	temp (°C)	method	type of dough	<i>K</i> (Pa-s″)	n (dimensionless)	R^2
bread mix ^a	50	36–38	on-line slit die	nonleavened control	250	0.49	0.8
				SCFX-leavened	128	0.63	0.9
flour-water ^b (9)	50	not reported	off-line slit die	nonleavened	1309-1390	0.46-0.48	>0.9
flour-water ^b (12)	64.5	21	off-line capillary die	nonleavened	1615-3115	0.24-0.46	>0.9
flour-water ^{b,c} (10)	45	30	off-line capillary die	nonleavened	1090-405	0.45-0.63	>0.9

^a Bread dough formulation described under Materials and Methods. ^b Mixture developed to 500 BU (9, 10, 12). ^c 3–60 min of mixing time, no rest time (10).

The consistency coefficient of the nonleavened control dough evaluated was lower than those reported in the literature (Table 2) because our extrusion temperature (36-38 °C) was higher than in previous studies (10, 12) (21 and 30 °C, respectively) and was equivalent to the proofing temperature of conventional bread dough to guarantee adequate dough development for good gas-holding capacity. Also, previous studies on extruded dough rheology (9, 10, 12) involved just flour-water mixtures, whereas our feed mix contained other ingredients, especially bakery fat, which is expected to have a lubricating effect. A one-to-one comparison is not possible, because our results represent an absolute on-line rheological characterization of dough during continuous production. The SCFX-leavened dough had a lower consistency coefficient than its nonleavened control, which suggests that SC-CO₂ acts as a diluent as well as an energy absorber.

The differences in apparent viscosity between the SCFXleavened dough and the nonleavened control dough tend to be more pronounced in the low shear rate region, whereas at high shear rates, the apparent viscosities tend to be similar (**Figure 4b**). This shows that the diluent effect of SC-CO₂ is more influential than the energy-absorbing effects in the low shear rate region, whereas it becomes less effective in the high shear rate region. An increase in material volume through the die causes the bubble nuclei to deform more and thus absorb more energy to be able to go through. Therefore, with an increase in shear rate, deformation of bubble nuclei starts to affect the rheological character of the product and tends to dominate above a wall shear rate level of 38 s⁻¹ (**Figure 4**), suppressing the diluent effect.

The above results support the fact that application of the novel SCFX technology rendered continuous production of a yeastleavened dough analogue possible, but additional baking studies are important to implement the overall quality of this half product on the road to the finished product, the bread. SCFX technology offers an attractive alternative for yeast-leavened dough production because it results in a substantial decrease in processing time (4-5 min), and further investigations on the final product are under way. Furthermore, SCFX technology alleviates emission problems by eliminating fermentation. It also offers the additional possibility of obtaining novel leavened dough alternatives because it is known to create unique microcellular structures and has flavor encapsulation capabilities. However, besides its attractiveness and application potential to the baking industry, the overall cost and process analysis, additional equipment costs, and additional product alternatives still need to be investigated.

A derivative benefit of the application of SCFX technology to leavened dough processing has been the fact that it renders absolute on-line rheological characterization of both nonleavened control and SCFX-leavened dough possible by allowing lowtemperature extrusion processing and on-line integration of CO₂. The continuous nature of the process and the possibility of creating a new generation of microcellular leavened dough of unique flavor characteristics make the process very attractive for industrial use. Additional work is indeed needed to enhance and improve the understanding of the process.

ABBREVIATIONS USED

BU, Brabender units; CAA, Clean Air Act; DATEM, diacetyltartaric acid esters of mono- and diglycerides; EPA, U.S. Environmental Protection Agency; SC-CO₂, supercritical carbon dioxide; SCFX, supercritical fluid extrusion; VOC, volatile organic compounds; WPC-34, whey protein concentrate.

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