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Impact of the use of unmalted adjuncts on the rheological properties of beer wort

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Abstract

Understanding the behavior of fluids is important in industrial processes of the most varied types. Changes in the composition of the brewing wort can have an impact on the phenomenon of transport of matter, important in the manufacturing as well as in the sensory perception of palatability and dietary characteristics. Changes in viscosity are also of interest to the industry, as this parameter plays an important role in the filtration theory. The present study intended to analize the impact of different brewing inputs on rheological parameters, using the same beer formulation as the basis, the variations being compared to the control. The adjuncts tested were rye, unmalted barley, and sorghum. The analizes were performed in an oscillatory and rotational rheometer, obtaining measurements from three moments of the beer production process. The main results are the differences in the flow curve profiles, showing greater pseudoplasticity when using rye, and that the formation of gel at high shear rates can be a frequent cause of problems in the brewery. This work can contribute to the creation of a methodology that allows maximizing the contents of dietary fibers without compromising the smooth running of the production process.

Keywords: brewing technology; rheometry; hydrocolloids; rheology.

Practical Application: This work can be applied in the development and scaling up of brewing formulations that intend to use, for reasons that can range from obtaining different sensory characteristics to some dietary properties, unmalted inputs in order to avoid critical conditions that affect the production of the wort.

1 Introduction

The extract that makes up the brewing wort can be obtained from malted and non-malted cereals. In addition to barley malt, the most common, wheat and rye malt and, to a considerably lesser extent, maize, sorghum, and oats are used (Kinze, 2019; Lima, 2010). Today malt is still one of the main brewing inputs, but to reduce costs or obtain a product with different sensory characteristics the use of unmalted adjuncts occurs in 85-90% of the beers in the world (Annemüller & Manger, 2013) and the growth in the consumption of craft beers has increased the demand for non-traditional ingredients (Betancur et al., 2020) that continue to have their uses studied (Zdaniewicz et al., 2020).

Some cereals, when non-malted, have large amounts of non-starch polysaccharides (NSP). NSPs in foods influence rheological properties, texture perception and are sources of dietary fiber. The two main groups of NSP are β-glucans and pentosans, which are associated with undesirable effects such as difficulty in wort separation and formation of turbidity (Autio, 2006).

Currently, adjuncts are described as any source of an extract except malt (Yemata, Wube, 2015) (Briggs et al., 2004). The use of adjuncts often increases the viscosity of the wort [8]. A 20-40% substitution of barley malt for oats increases the content of β-glucans and significantly decreases the performance of filtration and extract recovery. However, β-glucans make the beer more full-bodied (Yu et al., 2020) (Schnitzenbaumer et al., 2012). The use of rye is associated with production problems due to the high content of pentosans (Glatthar et al., 2005).

Among the changes in composition with impact in the process, the presence of β-glucans and pentosans is the most significant, which can cause viscous wort, slow wort separation, decreased extract recovery, slow filtration, and greater use of filters (Steiner et al., 2012). Aqueous solutions of different hemicelluloses (a type of β-glucan present in cereals) can form gels, and rheometric determinations obtained a pseudoplastic profile with yield stress, demonstrating a non-Newtonian flow behavior (Martínez-Ávila et al., 2014)

Newtonian and pseudoplastic flow behaviors have been observed with or without yield stress in the beer wort (Trávníček et al., 2015; Severa et al., 2009). This variability is due to differences in the wort extraction technique as well as in its formulation and analytical techniques. The concentration of sugars, the content of β-glucans and pentosans among other compounds have an impact on the flow profile of the fluid as well as on gel formations. It has already been pointed out that the application of shear stress guides the β-glucan molecules, making them align and form intermolecular interactions, forming gels (Bogdan, Kordialik-Bogacka, 2017). Rye has problems of filtration and wort separation widely described related to a greater deficiency of pentosan depolymerizing enzymes, an important NSP of this cereal. Solutions with higher levels of β-glucans cause a reduction in the Power Law index, an exponent of the Power Law model, indicating that its concentration is proportional to pseudoplasticity. Centrifugal clarification can cause gel formation due to the application of shear stress (Autio, 2006).

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In the nutritional approach, β-glucans and pentosans are dietary fibers. In the case of beer, 99% of the fibers are fermented by Lactobacillus and Bifidobacterium (Preedy, 2008). It has also been pointed out that these compounds may have prebiotic potential, and that their presence in beer is hampered by the lack of interest due to the associated manufacturing problems (Bamforth, 2005). Intake of NSP is also inversely associated with the development of metabolic syndrome (Chen et al., 2017) and soluble β-glucans have already been shown to reduce cholesterol in humans and mice (Anderson, 1990). Anyway, there may be a correlation of components that impact beer rheology with sensory and dietary aspects.

The objective was to analyze the impact of different brewing inputs on rheological parameters, using the same beer formulation as the basis, the variations being compared with the control.

2 Materials and methods

2.1 Inputs, equipment, and sample preparation

The brewing inputs used in the formulation of the control were: 100% malt Pale Ale - Muntons (Stowmarket, United Kingdom); hops Citra– Barth Haas (Germany); potable water. Adjunct formulations were: 80% malt Pale Ale - Muntons (Stowmarket, United Kingdom); 20% adjuncts (unmalted barley, rye, and sorghum); hops Citra - Barth Haas (Germany); potable water. The worts were prepared in 600 mL volumes in a conventional 304 stainless steel boiler Tramontina 2L (Carlos Barbosa, Brazil). An industry sample was also collected from a local brewery (PDC, São Paulo, Brazil) for having a total of 30% of unmalted adjuncts (unmalted wheat and oats).

The adjuncts were chosen by crossing three main criteria: the presence of NSP or other components that have an impact on beer production described in the literature; motivation to obtain certain organoleptic characteristics (ex: "full-bodied" beer); economic interest.

The base used to prepare the beer was the Double IPA style (Beer Judge Certification Program, 2015). The wort preparation followed the steps shown in the flowchart (Figure 1), based on a standard production technique (Kunze, 2019). Three samples of each wort were collected. The first and third samples had the same extract, measured by a refractometer in Brix degrees, one of the primary wort without hops and the other hopped and boiled wort. In this way, it is possible to evaluate the impact of adding hops and boiling on the rheology of worts. The second sample has a 20% dilution concerning the others, to check the influence of higher or lower concentrations of sugar in the wort rheology.

2.2 Rheometric analysis

The rheological characteristics of the wort were determined in a MARS Haake II rheometer with the RheoWin3 program for data analysis (Thermo Electron Corporation, Germany), the temperatures controlled by a refrigerated bath, and the sensor used was parallel plates type (PP60 Ti).

Each of the three formulations had three samples, two of the primary wort with the extract of 20° Brix and 16° Brix, and one of the secondary wort at 20° Brix. Each of these samples was submitted to rotational and oscillatory rheometric analyzes in, at least, triplicate. All tests were repeated at temperatures of 5 °C and 15 °C, except for sorghum that was tested only at 5 °C.

The rotational assay was performed in the Controlled Stress (CS) mode, from 0 to 0.5 Pa. This mode was chosen due to the ability to analyze a range with lower values of shear rate. The choice of the parallel plates measuring geometries was defined by the presence of particulate material that causes problems in measurements using a cone-plate sensor, in addition to being able to be used for measurements at low shear rates of fluids that have yield stress. The larger diameter compared to

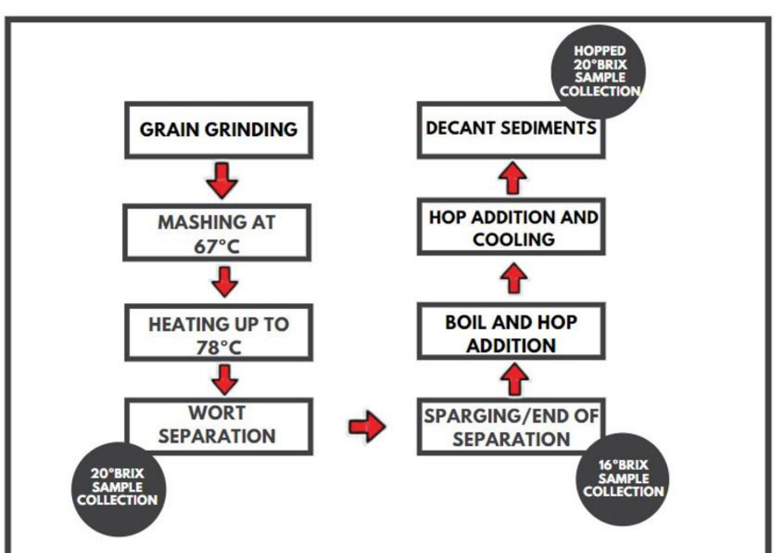

Figure 1. Production flowchart of beer wort with sample collection times.

some sensors allows a higher resolution of low viscous liquids (Schramm, 2006). The gap space between the parallel plates, was 0.105 mm, always adjusted before each analysis, corresponding to a volume of approximately 0.3 mL of wort

The oscillatory analyzes used the same measuring geometry, also in CS mode. First, the worts were subjected to an amplitude sweep analysis from 0 to 1 Pa and with a frequency fixed at 1 Hz. In this test is possible to identify if the fluid has an interval of linear viscoelasticity (LVE) and what is the limit amplitude for storage and loss modulus (G 'and G' ') to invert their position. If the wort had an LVE interval, a frequency sweep assay from 0 to 100 Hz with an amplitude set at 0.02 Pa was performed to obtain information about the microstructure of the formulation.

2.3 Statistical analysis

Data treatment and statistical analysis were performed using Origin 8 software (USA) and RheoWin3 program (Thermo Electron Corporation, Germany).

Linear and non-linear regressions to fit experimental data to mathematical models were the first steps for data analysis. The CS tests generate shear stress by shear rate graph, called a "flow curve". The results were subjected to linear and non-linear regression using the method of minimizing the sum of squares of the residuals. The nonlinear regressions had the Power Law as the starting model, with and without linear coefficient, since the Herschel-Bulkley model predicts the coefficient for considering the yield stress. If the linear model has the best fit in the results the wort can be described as a Newtonian fluid/Bingham plastic model: $\tau = \tau_0 + \gamma.\eta$ (τ is the shear stress, τ_0 is the yield stress, γ is shear rate and η is the viscosity). If it fits more to the non-linear regression, it can be described as pseudoplastic: $\tau = K(\gamma)^{\eta'}$ in simple Power Law model or $\tau = \tau_0 + K(\gamma)^{\eta'}$ in Herschel-Bulkley model (τ is the shear stress, τ_0 is the yield stress, γ is shear rate, η' is the Power Law index and K the index of consistency). The presence of a positive linear coefficient, cutting the ordinate axis, indicates that the fluid has yield stress below which it does not flow.

Although linear regressions normally use the coefficient of determination R^2 it can often provide statistically weak data for non-linear regressions in addition to the coefficient of determination R^2 tending to increase when the number of predictors is changed (Meyers et al., 2012), and the model Herschel-Bulkley's has one more predictor than the others. Therefore, it was decided to use the highest value of F, calculated from the division between the Average Squares of the model (Asm) and the Average Squares of the residuals (ASr) or simply F = ASm /ASr. The p-value is used to accept or reject whether the model fits the results, and the adequacy was accepted if Fcalculated > Ftablished at the 1% significance level. To compare the F values, each set was subjected to the paired t-test at a 5% significance level.

Even high values of F can be ambiguous to define whether the model fits the experimental data, so the residual analysis was used as an exclusion criteria for the model's validity. The methods used were the analysis of standardized residues and the analysis of the "residual x data order" graph. Residuals (ei) are standardized (di) by the following calculation: : di = ei /√MSr with the MSr being the mean square of the residues. If the standardized residual of any regression has a value not contained in the range [-3.3] it indicates the presence of an outlier (Meyers et al., 2012). If the residuals have a high degree of randomness, it can be assumed that the residuals are independent, and the model is well adjusted. If any systematic behavior is observed, we have indications that some other variable influenced the results of the experiment, which violates assumptions of the analysis of variance.

3 Results

The research design allowed comparisons between a control wort made only with malt and worts made with 20% of adjuncts. The influence of some parameters on the rheological characteristics could also be evaluated, such as temperature, wort concentration, and addition of hops/boil.

3.1 Rotational measurements

The visual analysis of the flow curve graphs (Figure 2) shows that, for the same concentration and temperature, the maximum shear rate of the worts with adjuncts is lower than that of the control with the application of equal stress. Also, in some tests, it was possible to observe a disturbance at high shear rates. The flow curves of both samples at 20 ° Brix showed a profile similar to that of Figure 2.

The proximity between the experimental data and the model data are shown in Table 1, expressed in F-value, with all regressions having a good adjustment at the 1% significance level (p = 0.01). The higher F, the more adjusted to the experimental data is the model. The Newtonian model, even with the linear coefficient to consider the yield stress parameter, did not present the best fit in any sample. The Power Law and Herschel-Bulkley models shared the best adjustments in the range of shear rates worked. The global difference between the worts adjustment averages, calculated from the t-test, showed that at 5 °C the Power Law and Herschel-Bulkley had no significant differences in adjustment at the level of significance α = 5%, and both significantly different from Newtonian regression at the same level of significance. At 15 °C the Power Law model was statistically better adjusted than Herschel-Bulkley and this, in turn, significantly more adjusted than Newtonian.

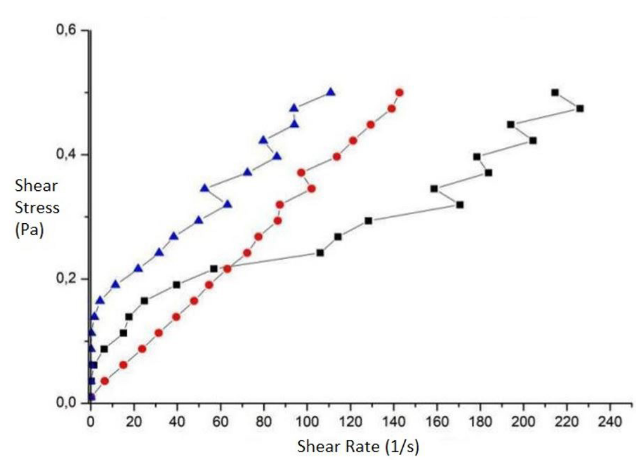

Figure 2. Wort flow curves. 100% malt control (squares), rye (triangles), unmalted barley (circles) 16° Brix at 5 °C.

Table 1. F-value (calculated) of the analysis of variance between the model and the experimental data for the worts.

		Newtonian		Power Law		Herschel-Bulkley	
		F 5 ºC	F 15 ºC	F 5 ºC	F 15 ºC	F 5 ºC	F 15 ºC
Control	**20º brix**	544	136	1138	790	1417	499
	16º brix	359	139	711	570	544	359
	20º brix hopped	1006	690	2400	1324	2563	1132
Rye	**20º brix**	330	405	1221	1665	1198	1461
	16º brix	284	2257	609	3679	635	2874
	20º brix hopped	294	1535	646	2199	611	2398
Barley	**20º brix**	486	2575	813	3494	674	3213
	16º brix	268	875	463	1827	500	1211
	20º brix hopped	571	986	1286	2929	992	1903
Sorghum	**20º brix**	684		1258		1165	
	20º brix hopped	1483		957		1896	
T-test α < 0.05	**5 ºC**	Herschel-Bulkley = Power Law > Newtonian					
	15 ºC	Power Law > Herschel-Bulkey > Newtonian					

Table 2. Power Law index values (η’) for the worts at temperatures of 5 and 15 °C obtained in non-linear regressions.

		Power Law		Herschel-Bulkley	
		η’ 5 °C	η’ 15 °C	η’ 5 °C	η’ 15 °C
Control	**20º brix**	0.505	0.342*	0.692	0.326*
	16º brix	0.523	0.389*	0.71	0.390*
	20º brix hopped	0.649	0.62	0.783	0.781
Rye	**20º brix**	0.419	0.486	0.563	0.654
	16º brix	0.348	0.859	0.59	0.962
	20º brix hopped	0.36	0.69	0.589	0.86
Barley	**20º brix**	0.682	0.866	0.86	1
	16º brix	0.309	0.791	0.634	0.869
	20º brix hopped	0.655	0.739	0.779	0.78
Sorghum	**20º brix**	0.588		0.765	
	20º brix hopped	0.745		0.952	

*model poorly fitted by the analysis of residues.

Considering that the Power Law and Herschel-Bulkley models showed greater adjustments concerning the experimental data, Table 2 contains the values of the exponent, called the Power Law index (η’), obtained in the regressions. The closer to 1, the smaller the concavity of the curve, and the closer to the line (closer to the Newtonian model) the flow curve approaches.

At 5 °C the control has a Power Law index different from 1, showing some degree of non-Newtonian behavior both in the Power Law model and in Heschel-Bulkley. Rye wort had an even lower η’, and barley and sorghum quite similar to the control in the Power Law model. Considering the Power Law indexes obtained in the Herschel-Bulkley regression, the rye wort remains with a lower η’ while barley and sorghum have higher values than the control, thus being closer to the Newtonian flow behavior. Comparing the rye and barley worts between the temperatures of 5 °C and 15 °C the value of η’ increased. In the samples 20° Brix and 16° Brix of the control at 15 °C, at high shear rates, the noise obtained in the curve (possible formation of a gel), which made it impossible to perform adequate regressions according to residual analysis.

Considering the Newtonian model (considering the yield stress), τ_0, as a Bingham plastic) for comparison, the parameters of each wort are described in Table 3.

In all cases, it is possible to deduce that the viscosity (η) is proportional to the wort concentration. Rye and barley worts were more resistant to flow than the control, except for the no hopped 20° Brix barley wort at 5 °C in which the shear rate (γ) was higher.

Analyzing Table 3, at 5 °C, the average viscosity of worts with 20° Brix (primary and secondary) was similar, with the rye viscosity being 4% higher than the control viscosity and barley wort was up to 5% smaller. Only sorghum wort showed viscosity lower than the control, about 50% lower. However, with 16° Brix worts, the adjuncts showed higher viscosity, with rye wort being 116% higher than the control and barley wort 93% higher. At 15 °C, they were also shown to contribute more to the viscosity. Whereas in the average of worts with 20° Brix, the viscosity of rye wort was 85% higher than barley wort, 63% higher. In samples with 16° Brix at 15 °C, rye wort had a viscosity 172% higher and barley wort 101% higher than

Table 3. Values of the parameters obtained in the linear regression (γ for shear rate, τ_0 for yield stress and η for viscosity) for the worts.

		η (mPa.s)		τ0 (mPa)		γ to 500 mPa (1/s)	
		5 °C	15 °C	5 °C	15 °C	5 °C	15 °C
Control	**20° brix**	4.05	1.28	82	145*	102	254
	16° brix	1.77	1.13	74.6	105*	214	337
	20° brix hopped	4.65	2.60	61.2	63.7	95.3	162
Rye	**20° brix**	4.57	3.52	95.5	85.8	84.6	122
	16° brix	3.83	3.08	99.3	27.4	110	148
	20° brix hopped	4.52	3.66	96.9	54.2	83.8	120
Barley	**20° brix**	3.81	3.38	55.9	27.7	121	140
	16° brix	3.14	2.28	14.0	37.2	150	205
	20° brix hopped	4.48	2.96	60.6	45.6	103	151
Sorghum	**20° brix**	2.26		68.2		193	
	20° brix hopped	2.17		56.4		208	

*model poorly fitted by the analysis of residues.

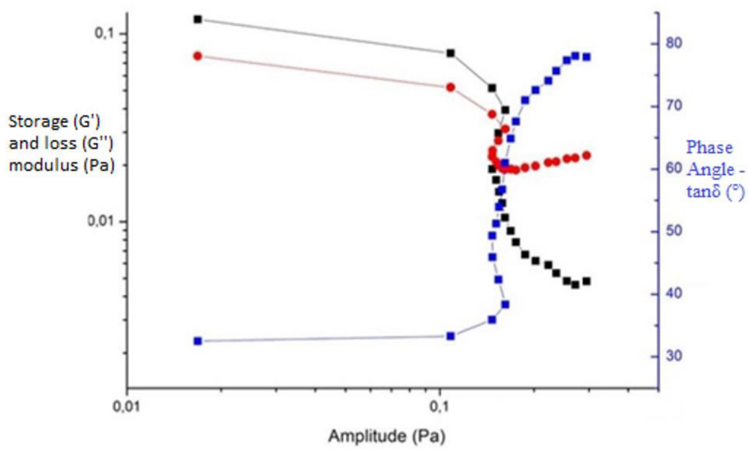

Figure 3. Stress sweep of the control sample 20° Brix at 5 °C. Storage modulus (G' - black squares), loss modulus (G" - circles) and phase angle (tanδ - blue squares).

the control. Comparing the primary wort with the secondary, whose concentrations are 20° Brix, the viscosity values do not present significant differences (α = 0.05).

The effect of temperature on the yield stress (τ_0) is not conclusive. In the control, this value was higher for the temperature of 15 °C than 5 °C. However, this is due to a bad adjustment of the first points of the regression, verified by a linear trend at the beginning of the graph "residual x order of data collection". However, the most relevant data is that the yield stress values are always very low. For comparison, the yield stress of Ketchup is close to 15 Pa (Schramm, 2006), more than 150 times the average of the values measured in this work.

3.2 Oscillatory measurements

The SS analysis, or stress sweep, outputs a graph showing the storage and loss modulus (G 'and G' ') as well as the phase angle, defined as tanδ = G"/ G', given in degrees. In all samples, it was possible to observe the inversion of the modulus (Figure 3). At low stress, the storage modulus is larger than the loss modulus, indicating a higher elastic behavior than viscous. As the stress increases, the modulus is inverted, and the phase angle begins to increase.

Also, in Figure 3, it is possible to observe the linear viscoelasticity interval, in which the phase angle does not change with increasing amplitude (stress). This phenomenon occurred in samples on a small scale, with a small linear viscoelasticity interval. However, the linear viscoelasticity interval of the industry sample collected (Figure 4) presented a classic curve with an inversion of modulus with higher stress. This sample contained 30% of adjuncts, 50% more than those formulated in the laboratory. At high stress, the samples showed small differences between the viscous and the elastic modulus, with an inversion of the initial property (predominantly elastic). The linear viscoelasticity interval of the industry sample occurs at 20° of phase angle, about 10° less than that shown in Figure 3, showing that the increase in adjuncts resulted in wort with a larger elastic component (closer to an ideal solid). Another big difference between the results of the worts produced in the laboratory and the wort of the industry is the values of the modulus in the first plateau of linear viscoelasticity and the second: in Figure 3 it is possible to see that G ‘ goes from approximately 0.1 Pa to 0.005 Pa (20 times less) and the G ‘’ starts at approximately 0.08 Pa and reduces to about 0.025 Pa (3.2 times less). The scale difference in Figure 4 is extremely larger, going from about 9000 Pa to 0.05 Pa (180 thousand times less) for G ‘and from 3000 Pa to approximately 0.15 Pa (20 thousand times less) in G ‘’. This indicates that a deeper transformation from a predominantly elastic to a predominantly viscous character that occurs when the content of adjuncts in the formulation is increased and that the use of adjuncts in this sample generated a fluid with greater structure. It was not possible to place the graphs on the same scale without impairing the observation of the data.

The last was the frequency sweep (FS) tests. The complex viscosity shown in Figure 5, decreases with increasing frequency, showing the pseudoplasticity of all wort formulations up to approximately 10 Hz. However, at frequencies even greater than 10 Hz, there is an inversion, and the complex viscosity rises abruptly, indicating the occurrence of a possible shear gel formation. It is also possible to notice that the rate of variation of the complex viscosity in the control was lower, indicating that the adjuncts influence the non-Newtonian behavior of the wort. Storage modules always have higher values than loss modulus (except at one point on the control), and the more pronounced slope of the pure malt control indicates less fluid structuring force.

4 Discussion

Despite the generation of mathematical models to visualize and analyze the flow behavior of formulations containing different adjuncts, a considerable limitation of the study is the lack of data on the composition of these cereals. The cultivars have differences in their composition according to variables such as soil, harvest time, rains, etc. that can cause the same adjunct to present different results according to producer/region/variety (Lima, 2010). The increase in the fluidity and flow profile is related to specific components of cereals, such as NSP and

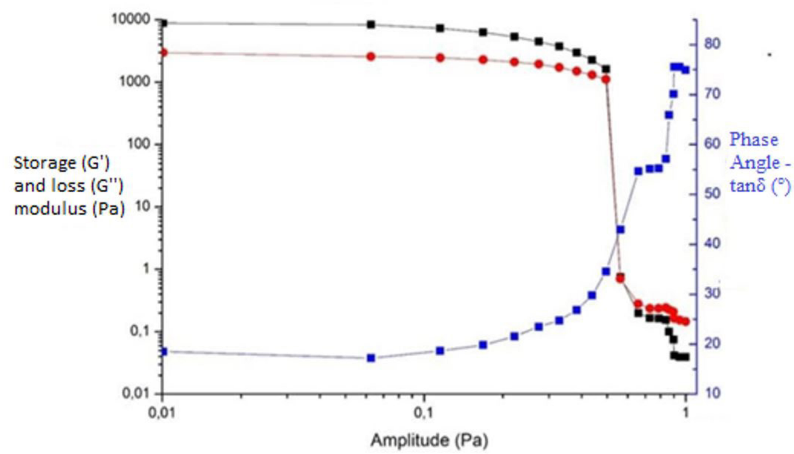

Figure 4. Stress sweep - Industry wort sample (30% of adjuncts, unmalted oats, and wheat) at 5 °C. Storage modulus (black squares), loss modulus (circles) and phase angle (tanδ - blue squares).

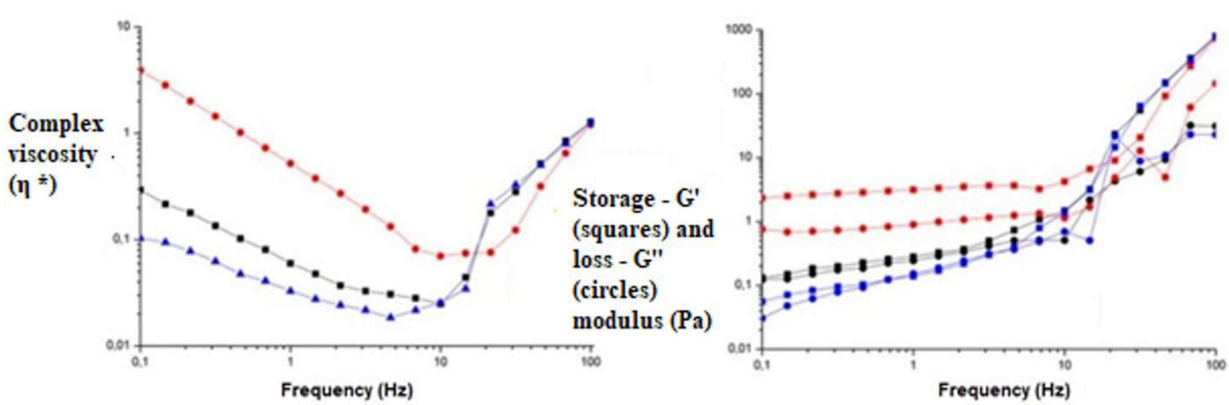

Figure 5. Complex viscosity (η *) and storage and loss modulus obtained in the Frequency Sweep assay on wort samples with 20° Brix at 5 °C. Control wort (triangles on left and blue on the right), unmalted barley (circles on the left and red on the right) and rye wort (squares on the left and black on the right).

proteins (Autio, 2006). Thus, for a deeper understanding of the components that alter the flow, quantitative relationships between the centesimal components and viscosity would bring more accurate information.

Based on the flow curves of rotational tests, it is possible to state that at low shear rates, the behavior of worts can be described as non-Newtonian, as previously observed in pure malt beer worts (Trávníček et al., 2015) (Severa et al., 2009). Adjuncts influenced viscosity by increasing it, resulting in a lower shear rate for the same stress. Rye wort had the effect of increasing the concavity of the curve, expressed in values of η', further distancing it from Newtonian behavior. Unmalted barley had no significant effect on η', only on dynamic viscosity (η). The wort with sorghum showed lower viscosity and a higher Power Law index η', indicating that its rheological characteristics favor the transport of matter and processing.

The results of the oscillatory tests offered a better understanding of observations resulting from rotational tests. The increase in the phase angle in all samples shows pseudoplasticity in the beer wort. The wort of the industry, which had a higher percentage of adjuncts, presented a larger LVE interval compared to the other samples. In the frequency sweep it was possible to observe a tendency of the viscous characteristics to surpass the elastic ones with the increase of the frequency from 0 to 10 Hz, however from 10 Hz there is an inversion with an abrupt increase in the complex viscosity, indicating cross-linking of the structure and possible gel formation like observed in centrifugal clarification (Autio, 2006)

Considering the Newtonian model, it was found that the worts have very low yield stress values if compared with other fluids like ketchup.

Rheometry can be an important tool both in the development of new beers and in quality control. Due to the interest in producing beers with adjuncts (Annemüller & Manger, 2013) either for motivation to obtain a different flavour or to provide soluble dietary fibers and phenolic compounds, one can test the gel formation on a small scale to know the maximum content of adjuncts without cause compromise of the process. As the grains present different levels of NSP according to crop, cultivar, region, among other parameters, an industry that has changed suppliers can check the quality of the grains and plan production using rheometric tools. The tests can give information about the structure of the wort on a small scale, facilitating a conscious and planned up-scaling that will avoid problems in large dimensions, which are usually costly. Sorghum does not have large amounts of NSP (Autio, 2006) and the rotational tests showed that the use of this adjunct in the 20% content resulted in a high processability wort (low viscous, low pseudoplasticity, and without gel formation observed in rotational tests) and that its use, now practically restricted to the African continent, can be further explored.

The parameters obtained in rheometric tests can be used as quality control of the wort. In routine analyzes of quality control of malts and brewing adjuncts in the industry, the only rheological test that is performed is the kinematic viscosity of the primary wort, a point measure obtained in a capillary viscometer, being insufficient for more complete understandings about structural behavior when working with new raw inputs.

5 Conclusions

Differences between worts made with different formulations, containing or not containing adjuncts, were possible to establish. The pseudoplasticity of the wort prepared with rye was greater than the control, while the sorghum wort showed a behavior closer to Newtonian more than the control, both evaluated by the Power Law index (η'). The number of adjuncts, when compared to the control sample and to the industry sample showed to be important in increasing the elasticity and the linear viscoelasticity interval.

Processability, which means fast separation, clarification, and filtration of the wort and without clogging, is inversely proportional to the viscosity, size of the LVE interval, pseudoplasticity (according to the Power Law index η', exponent in the Power Law model), and to the complex viscosity, justified by the differences found in the evaluated worts. The gel formation observed in some results of this work is a property that needs to have its impact even better elucidated, mainly in production. In other words, dynamic viscosity parameters may be more interesting than kinematics to assess the behavior of the brewing wort.

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