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Research Article

# TEMPERATURE DEPENDENT DYNAMIC OSCILLATORY RHEOLOGICAL CHARACTERIZATION OF BARNYARD MILLET FLOUR

**NISHA R.[1], PANDIARAJAN T.*[1], GANAPATHY S.[1], AMIRTHAM D.[1], DURAISAMY M. R.[2], ARULMARI R.[1]**
[1]Department of Food Process Engineering, Tamil Nadu Agricultural University, Coimbatore, 641003, India
[2]Department of Physical Sciences and IT, Tamil Nadu Agricultural University, Coimbatore, 641003, India
*Corresponding Author: Email - pantry_apk@yahoo.co.in

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**Abstract:** Rheology concerns with the flow and deformation of substances and, in particular, their behaviour in the transient area between solids and fluids. Rheologicl behaviour of barnyard millet four was studied under dynamic shear studies. The millet flour was set for three different temperature *i. e* 80, 100 and 120˚C for which dynamic shear and pasting properties of the flour was studied, the highest Pasting temperature Viscosity (cP) for 80, 100 and 120ºC were 100.2, 122.3 and 330.6. Storage and loss modulus G' and G'' and flow behaviour were studied. Results indicated that storage modulus was higher than loss modulus at all temperature and the best fit model was Herschel- Bulkley.

**Keywords:** *Barnyard millet, Flow behaviour, Storage modulus, Loss modulus, Pasting profile*

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## Introduction

Barnyard millet is one among minor millet; this nutrient rich millet has high fibre content, which can effectively help in losing weight. It is a rich source of calcium and phosphorous, which helps in bone building and its daily consumption helps in fighting against bone diseases. Presently people are opting for millets due to its health benefits and as an alternative for rice and wheat. Millets are utilized in industrial scale for manufactured of food like snack food, dietary food *etc*. During processing of food like extrusion cooking, gelling, baking will be fully dependent on proper gelatinization of starch [1]. Dispersing the cereal flour in water depend on suspension rheology which mainly depends on starch content, its gelatinization, soluble and insoluble solids with the temperature measurements [2]. Presently there are very literatures on the flow properties of the flour and its behavioural characterization. Dynamic oscillation measurements can be measured in terms on storage and loss modulus, which gives us information on the stored energy and the energy dissipated as heat [3]. Dynamic test are mainly performed by applying small sinusoidal strain/stress and measuring resulting stress/ strain. These small-amplitude oscillatory tests are commonly performed in shear and hence the abbreviation SAOS, for small-amplitude oscillatory shear, is commonly used to represent dynamic viscoelastic tests [4] Though not yet very popular, the dynamic tests are also performed in compression/tensile mode and in large amplitude shear mode (at strains in excess of 100%). It is important to emphasize that the strains (and stresses) used in SAOS tests are very small, often in the order of 1 to 3 or 5%. This is to assure that the material response is in the linear range the range within which the stress is proportional to the applied strain, and the theory described below is applicable. The focus of this paper is to present only the linear viscoelasticity as measured by the SAOS tests. The frequency dependent functions G' and G"are shear elastic (storage) modulus and shear elastic (loss) modulus, respectively [5]. G'is a measure of the energy stored and subsequently released per cycle of deformation per unit volume and. The property relates to the molecular events of elastic nature. G''is a measure of the energy dissipated as heat per cycle of deformation per unit volume. G'' is the property that relates to the molecular events of viscous nature.

### Sample preparation

Barnyard millet were cleaned to remove dirt and discoloured grains, washed with tap water and sun dried by placing on clean cloth. The sample was made into flour using a burr mill in a local flourmill to a 100-micron particle size that passed through ISS 40 metal sieve and cooled to room temperature to avoid clump formation and stored in airtight containers for further experiments. Calculated amount of flour and distilled water were taken to obtain the fresh paste. The slurry was prepared in the ratio of 5g of flour and 25ml of distilled water [6] The prepared paste was preheated in a water bath for about 25ºC to make the flour completely solubilized for paste formation before tarting the test. The prepared paste was then allowed to cool down to avoid crump formation.

## Dynamic measurements

### Flow behaviour

The study was conducted at a controlled shear rate (CSR) mode for the barynyard millet flour samples at three different temperatures, *i.e*., 80ºC, 100ºC and 120ºC considering in context with food extrusion cooking. Measurement were made using an upper movable parallel plate (PP 50) rheometery setup attached to measuring head and lower fixed base platform held along P-PTD 200/AIR has an integrated air heat exchanger. The freshly prepared paste was placed between the plates and experiment was carried out. The shear rate was increased from 0.01 to 100 $s^{-1}$ at varying shear stress at shearing with 25 measuring point were generated and each measurement were replicated [7]. The rheogram obtained from shear stress and shear rate values obtained were fitted four different rheological models that have software (Anton paar, MCR 52) though non linear regression analysis.

The models fitted were:

Bingham: $y = a + b \cdot x$
Casson: $y^{1/p} = a + b \cdot x^{1/p}$
Herschel-Bulkley: $y = a + b \cdot x^{p}$
Ostwald: $y = a \cdot x^{b}$

Where, y is the shear stress (Pa), x is the shear rate ($s^{-1}$), a is the consistency index (Pa $s^n$), b is the infinite shear viscosity (Pa s). The goodness of fit for the model-developed models was evaluated by determining the coefficient of determination R2 and standard deviation.

**Amplitude sweeps**

Amplitude sweeps at a constant frequency (10 rad/s) to determine the maximum deformation attainable by a sample in the linear viscoelastic range and Frequency sweeps at a constant deformation (0.5% strain) within the linear viscoelastic range. The dynamic moduli spectra would be G', G" and tan δ as a function of frequency. Rheologicaly, G' is the dynamic elastic or storage modulus, gives information on the material response as a solid. G" is the dynamic viscous or loss modulus gives information on material response as a fluid. The data of all rheological measurements were analyzed with the supporting software Rheoplus/322 v 2.81 with model Anton Paar MCR 52 model. Plate- peltier temperature device P-PTD 200/AIR was used which has a fixed lower measuring plate with peltier temperature controller that is suitable for MCR 52, P-PTD 200/AIR has an integrated air heat exchanger. The measuring system PP-50 was used for the study attached to coupling of the measuring head. The prepared sample placed on the lower plate of the P-PTD 200/AIR and moving plate was bought to measuring position of gap 0.5mm and the sample was then trimmed off. Each paste was transferred so that the residual stress would be released. The study was selected for oscillation mode and the test was carried out.

**Pasting properties**

A Modular compact Rheometer (MCR 52) attached with Starch cell (C-ETD 160/ST) was been used to determine the pasting properties of barnyard millet flour. Fresh paste of flour were prepared having 3.5g of the millet flour and 25ml of the distilled water kept in a stirrer for about 10 min for proper mixing of the flour and water. Test was carried out using electrically heated cylinder system with the fluid cooling which includes a tool master. Once the instrument is initialized the stirrer (ST 24) was firmly fix to the measuring head having MCR coupling and zero gap adjustment were made. The prepared samples were loaded in measuring cup (aluminium) and fixed for measuring; the cup is locked in the place with the handle. Once the tool master, starch cell and measuring cell is selected, set the fluid circulator to 15$^0$C and wait for thermal equilibrium. A refrigerated circulator consists of a refrigerated bath, controller, ventilation grid controlled the temperature and main cables with the recommended temperature of 5 to 95ºC, the filling level of the interior bath is maximum up to 2cm below the cover plate. Move the measuring position into the sample and start the measurement. For the given instrument the starch cell can be used to a temperature of 160$^0$C, the maximum heating rate of the starch cell is 60 K/min and maximum cooling rate is 45 K/min. The sample was equilibrated at 50$^0$C at 960 rpm for 5 measuring points, measuring point duration 2s and interval duration is 10s, then heated from 50 to 90$^0$C at 160 rpm for 225 measuring points, sample were held at 95$^0$C 160 rpm for 150 measuring points finally cooled 95 to 50$^0$C, 160 rpm for 22 measuring points. The viscosity profile recorded was a programmed heating and cooling cycle reflects the rheogram of the pasting temperature (PT), Peak time and viscosity (PV), holding strength (HS), breakdown viscosity (BD), Final viscosity (FV), set back from peak, set back from trough were obtained from instrument and all the determination were performed in triplicate And average values were recorded.

**Results and Discussion**

**Steady shear- Flow behaviour**

The flow behaviour for samples at three temperatures is shown in the fig. The slope of the line at 80 and 100 increase gradually and for 120ºC there was decline in the slope, for the three temperature the best fitted model was Herschel- Bulkley, Which is a generalized model of a non-Newtonian fluid, in which the strain experienced by the fluid is related to the stress in non-linear. The $R^2$ value was high in Herschel- Bulkley giving values of 0.93184, 0.9701 and 0.85661 for 80, 100 and 120ºC.

**Pasting profile of millet flour**:

Pasting temperature, which provides an indication of the minimum temperature required to cook a given sample, can have implications for the stability of other components in a formula, and also indicate energy costs. For the given sample the pasting temperature increased with increase in temperature, the highest pasting temperature obtained were 100.2, 122.3 and 330.6 (cP) for 80, 100 and 120˚C.

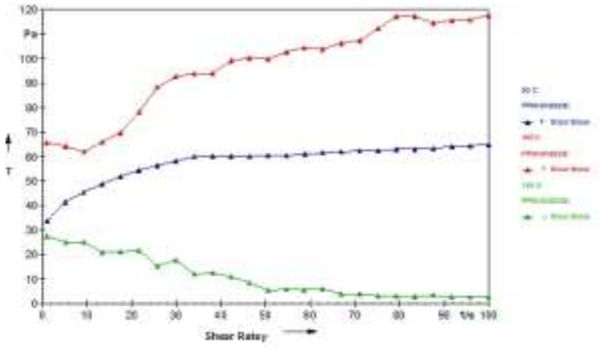


Fig-1 Flow curve for millet flour at 80˚C, 100˚C and 120˚C

Table-1 Estimated rheological constants and statistical parameters

| Model name | Model parameters | Temperature | | |
|---|---|---|---|---|
| | | 80˚C | 100˚C | 120˚C |
| Power Law | a | 37.698 | 34.034 | 945.99 |
| | b | 0.11903 | 0.27504 | -1.273 |
| | $R^2$ | 0.9201 | 0.95602 | 0.70742 |
| | SD | 1.1384 | 3.1361 | 3.6413 |
| Bingham | a | 51.492 | 66.938 | 13.832 |
| | b | 0.15131 | 0.58645 | -0.12791 |
| | $R^2$ | 0.78598 | 0.87005 | 0.55703 |
| | SD | 1.8632 | 5.3905 | 4.4805 |
| Casson | a | 6.7422 | 7.0069 | 6.4558 |
| | b | 0.13794 | 0.40979 | -0.51729 |
| | $R^2$ | 0.85862 | 0.92076 | 0.09205 |
| | SD | 1.5144 | 4.2093 | 1.8981 |
| Herschel- Bulkley | a | -649.82 | -2,516.4 | 880.39 |
| | b | 682.73 | 2,516.8 | -839.39 |
| | $R^2$ | 0.93184 | 0.9701 | 0.85661 |
| | SD | 1.0515 | 2.5856 | 2.5491 |

Peak viscosity indicates the water-holding capacity of the starch in the millet flour. It is often correlated with final product quality, and also provides an indication of the viscous load likely to be encountered by a heating. For the given sample the Peak viscosity increased with increase in temperature, the highest pasting temperature obtained were 337.6, 2,498 and 2,774 (cP) for 80, 100 and 120˚C. The rate of breakdown in viscosity to a holding strength depends on the temperature and degree of mixing, or shear stress, applied to the mixture, and the nature of the material itself. The ability of a sample to withstand this heating and shear stress is an important factor for many processes.

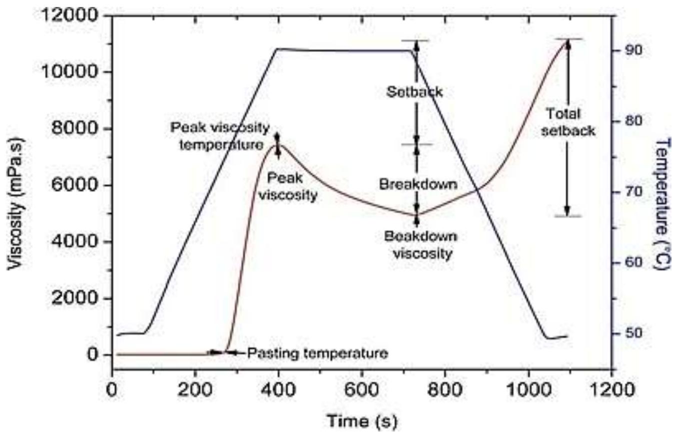


Fig-2 Starch pasting curve showing typical measurements

Table- 2 *Pasting profile*

| Variable Temp. | Peak time Viscosity (cP) | Pasting temperature Viscosity (cP) | Holding strength Viscosity (cP) | Breakdown Viscosity (cP) | Final viscosity (cP) | Setback from peak (cP) | Setback from trough (cP) |
|---|---|---|---|---|---|---|---|
| 120˚C | 2,774 | 330.6 | 323 | 2,451 | 347.7 | 2,427 | 24.73 |
| 100˚C | 2,498 | 122.3 | 813.6 | 1,684 | 2,454 | 44.03 | 1,640 |
| 80˚ C | 337.6 | 100.2 | 161.1 | 176.6 | 1,025 | -687.1 | 863.7 |

For 80 ºC, 100ºC and 120ºC the holding strength viscosity was 161.1, 813.6 and 323 (cP) and breakdown viscosity was 176.6, 1,684 and 2,451 (cP). The re-association between starch molecules during cooling is commonly referred to as the setback. It involves retrogradation, or re-ordering, of the starch molecules, and has been correlated with texture of various products. Set back from peak 80, 100 and 120ºC was -687.1, 44.03 and 2,427. Final viscosity is the most commonly used parameter to define a particular sample's quality, as it indicates the ability of the material to form a viscous paste or gel after cooking and cooling. Final viscosity 80, 100 and 120ºC were found to be 1,025, 2,454 and 347.7 (cP)

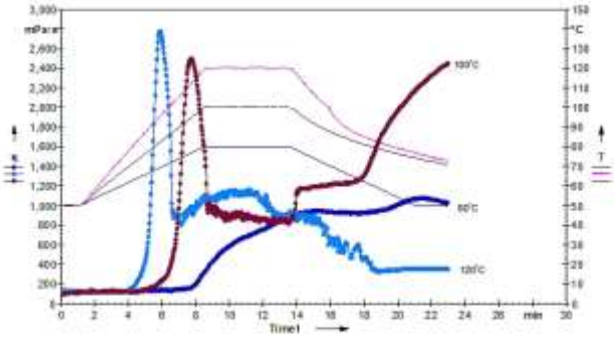

Fig-3 Pasting profile of the millet flour with varying temperature

**Amplitude sweep test for the flour at different temperature**:

Linear viscoelastic region (LVER) denotes the indicates the range in which the test is carried out without destroying the structure of the sample. It is the region shown on the left side of the diagram that is the range with the lower strain values. The below figure shows the variation of G' and G''with respect to increasing strain for the millet flour at different temperatures, for all the temperature variation the value of G' > G'' within the LVE region, thus the sample has a gel-like or solid structure. Value of the G' being greater than G'' at the initial stage, the samples displayed a narrow linearity range showing the material is soft. A constant linearity of the LVER was seen at temperature 80 and 100˚C than that of higher temperature. The crossover point, *i.e.,* G' = G'' was reached at 1% strain at 120ºC, 100% strain for 100ºC, and 90ºC for 80ºC, indicating that the spread of the sample and breakdown of the sample. At 10 % strain where the point is said to be cross flow point, due to gradual increase strain to the sample the material inner structure is breakdown and yield point reaches

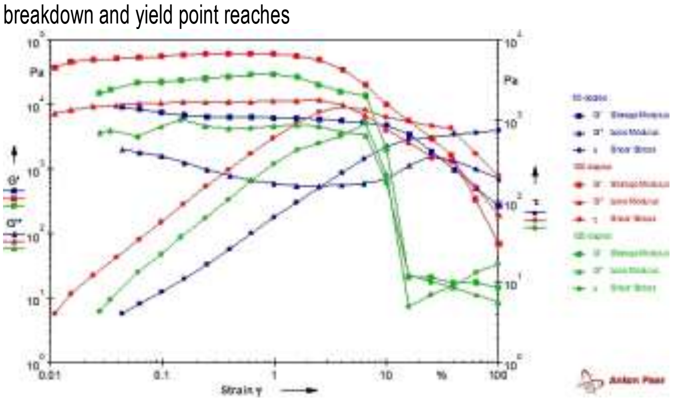

Fig-4 Dynamic viscoelastic properties of barnyard millet flour at different temperature

## Conclusion

The peak gelatinization temperature of barnyard millet flour were established, flow behaviour of millet flour for r all the three temperature were obtained, and fitted for all the non-Newtonian models *i.e.,* power law, Bingham, casson and Herschel-Bulkley and best fit was Herschel- Bulkley.

**Application of research**: Gelatination temperature of highest peak values were obtained, the storage and loss modulus values results showed the indication of the viscoelatic behaviour and yield point and cross flow of the flour were obtained.

**Research Category**: Food Process Engineering

**Abbreviations**: G'- Storage modulus, G''- loss modulus, η- Viscosity

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**Study area / Sample Collection:**

**Cultivar / Variety name:**

**Conflict of Interest:** None declared

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Ethical Committee Approval Number:

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