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ORIGINAL ARTICLE

# A study on rheological characteristics of roller milled fenugreek fractions

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**Abstract** Fenugreek seeds were fractionated by roller milling to get various fractions. The roller milled fractions and whole fenugreek flour (WFF) were evaluated for the flow behavior and time-dependent flow properties using a rotational viscometer at the temperatures of 10–60 $^0$C. The samples subjected to a programmed shear rate increase linearly from 0 to 300 $s^{-1}$ in 3 min and successive decrease linearly shear rate from 300 $s^{-1}$ to 0 in 3 min. The roller milled fractions and WFF paste exhibited non-Newtonian pseudoplastic behavior. Difference in hysteresis loop area was observed among the roller milled fractions and WFF, being more noticeable at lower temperatures. Power law and Casson models were used to predict flow properties of samples. The power law model described well the flow behavior of the roller milled fractions and WFF at temperatures tested. Except flour (FL) fraction, consistency coefficient, m, increased with the temperature both in the forward and backward measurements. The roller milled fractions and WFF exhibited rheopectic behavior that increased viscosity with increasing the shear speed and the temperature. For all the sample tested, initial shear stress increased with increase in shear rate and temperature.

**Keywords** Rheology · Fenugreek · Fractions · Flow behavior · Non-newtonian behavior · Rheopectic

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## Introduction

Fenugreek (*Trigonella foenumgraecum*) is an annual herb that belongs to the family Leguminosae and widely grown in India, Pakistan, China, Egypt, and Mediterranean countries. Fenugreek has anti-diabetic and hypercholesterolemic properties (Mondal et al. 2004; Vats et al. 2002; Rao 1996; Barbosa-Ca'novas et al. 1996; Venkatesan et al. 2003). The fenugreek seed contains higher proportion of protein content which ranges from 23 to 43 % (Taylor et al. 2000). Fenugreek seed protein contains unique free amino acid 4-hydroxyisoleucine, which is one of the active ingredients that have insulinotropic and anti-diabetic properties in animal model (Flammang et al. 2004; Haeri et al. 2009). Handa et al. (2005) in their studies showed that fenugreek extract enriched with free amino acid 4-hydroxyisoleucine reduced body weight gain induced by high fat diet. Fenugreek is a good source of dietary fiber and has 45 to 50 % dietary fiber, with 15–20 % of galactomannan, a soluble fiber. Lipid content of fenugreek seeds ranges from 5 to 10 % mainly consists of neutral lipids, namely, 6.3 % triglycerides and 450 mg/100 g phospholipids (Shankaracharya et al. 1973). The fenugreek seeds also contain micronutrients such as flavonoids, saponins, coumarins, and more calcium, phosphorous, iron, zinc, and manganese than in the most legumes (Schryver 2002).

India produces about 50,000 t of fenugreek and is one of the major producer and exporter of fenugreek. India claims 70–80 % of worlds export in fenugreek (Edison 1995). Fenugreek grain consists of outermost yellowish brown seed coat or husk and it is separated from the embryo by whitish endosperm, which is mainly composed of soluble galactomannan gum.

The roller milling process is a mechanical gradual grinding process where the endosperm is separated from the outer layers. Roller milling operation essentially involves scraping out and grinding of inner part of the grain from outer part, and sorting the particles by sieving into grades by size and degree of purity. The process is repeated several times with the objective of obtaining the maximum yields with clean separations of different parts of the grains. The roller milling process has a potential to produce various fractions of grains on milling with unique composition and functional properties (Sakhare et al. 2014a, b; Kiryluk et al. 2000). Recently, Sakhare et al. 2014b showed that the roller milling is a valuable process for fractionation of fenugreek to produce new and nutritionally valuable fenugreek products. The soluble fiber present plays a vital role in both in physical and nutritional properties of fenugreek fractions. Thus rheological studies were taken up for the roller milled fractions and compared with WFF.

Rheology is the study of deformation and flow of matter. The rheology has many applications in the fields of food processing, food handling and food acceptability (Barbosa-Ca'novas et al. 1996). The rheological data are required for process engineering analyses such as mixing, agitation, heating, extrusion, pumping, coating and process control. It is also required for quality control, shelf-life estimation, texture evaluation, product development and development of constitutive equations for rheological characterization (Ofoli 1990).The soluble fiber of fenugreek seed is glactomannan, which is made of two major sugars mannose and galactose like the other soluble fiber of guar seeds, psyllium husk etc. The soluble fiber is depressed throughout the seed coat and also found in endosperm (Madar and Shomer 1990). The most important property of galactomannans is their high water-binding capacity and the formation of highly viscous solutions at relatively low concentrations. The uniqueness of the fenugreek galactomannans composition is the content of galactose and mannose residues in the ratios of 1:1. The galactomannans from guar, locust bean and fenugreek are mainly used as thickening, stabilizing and emulsifying agents for foods (Brummer et al. 2003; Garti et al. 1997; Huang et al. 2001). They exhibit non-Newtonian behaviour in which the viscosity decreases with the increase in shear rate.

The objective of this study was to evaluate the rheological properties of roller milled fenugreek fractions for flow behavior and time-dependent rheological properties under different temperatures and shearing conditions. The suitability of the rheological models employed to fit the shear rate and shear stress data has also been evaluated.

## Materials and methods

### Materials and roller milling

Fenugreek seeds were obtained from the local market and used for the study. Fenugreek seeds were cleaned and water conditioned to a moisture content of 15.5 % and rested for 24 h before milling in the air tight container. The conditioned fenugreek samples milled using Buhler laboratory-scale roller mill Sakhare et al. 2014b. The Buhler laboratory mill includes six grinding stages and their corresponding sifting sections. The break rolls are fluted and reduction rolls are with smooth surface to produce flour. All products are conveyed by pneumatically. The laboratory bran duster was used for dusting of coarse and fine husk to remove any attached embryo portion. Six flours produced on the mill are combined to get the final flour (FL). Other milled fractions were coarse husk (CH), fine husk (FH) and pollard (PL). The whole fenugreek flour (WFF) and roller milled fractions were used for the study.

### Chemical composition

The roller milled fenugreek fractions and WFF were evaluated for moisture content, ash, protein, fat according to the standard methods (AACC 2000) and dietary fiber (AOAC 1999). All the values were calculated on a dry mass basis.

### Color measurements

The color of the roller milled fenugreek fractions and WFF was measured using the Hunter Lab color measuring system, Model Labscan, XE (Reston USA). The L a b color scale was used where L is the degree of lightness value, a is intensity of color in the direction of green to red color and b is intensity of color in the direction of blue to yellow. Samples were placed in the sample holder and the reflectance was auto recorded for the wavelength ranging from 360 to 800 nm.

### Sample preparation

WFF was produced by grinding the whole fenugreek seeds in the lab hammer mill and coarse roller milled fractions also milled using lab hammer mill to produce the flour of <250 μm. Aqueous solutions of roller milled fractions and whole fenugreek flour were prepared using a magnetic stirrer. The concentration level employed 3 % *w*/*v* for WFF, CH, PL, FH and 6 % *w*/*v* for FL. Measured amounts of test samples were dispersed into distilled water in instalments taking care to avoid any lump formation. The samples were added to vortex created in the water, taking care not to disperse the product onto the side walls of the beaker or on the stirrer paddle rod within 20–30 s. At the end samples were mixed at high speed for 5 min. The samples were assesed for the flow characterstics at diffrent temperatures (10, 20, 30, 40, 50 and 60 °C).

### Rheological measurement

Rheological properties of prepared samples were determined using MCR-52 rheometer 75 (Anton Paar, Graz, Austria) using a parallel plate geometry equipped with P75 measuring tool (diameter of 75 mm, gap of 1 mm) and used in the rotational mode and programmed via a computer controlled Rheoplus software (Anton Paar, Graz, Austria). After loading the sample, there was a 5 min waiting period to allow the sample to recover and achieve temperature equilibrium and samples were subjected to a programmed shear rate linearly increasing from 0 to 300 $s^{-1}$ in 3 min and successive linearly decreasing shear rate from 300 $s^{-1}$ to 0 in 3 min. Shear stress-shear rate data were gathered as a rheograms. In the second part of this study, the time-dependent rheological properties were investigated by shearing prepared fenugreek roller milled samples at different temperatures and three different constant shear speed.

### Data analysis and calculations

The flow curves of fenugreek roller milled fractions and WFF were modelled by using power law model, which is more engineering application:

$$\text{Power law model}: \sigma = \mathrm{m}(\gamma)^n \quad (1)$$

Where, σ is shear-stress (Pa), $\gamma$ is shear-rate ($s^{-1}$), m is consistency index ($Pas^n$), $n$ is flow behavior index (dimensionless).

$$Cassonmodel: \sigma^0.5 = (\sigma_c)^0.5 + kc(\gamma)^0.5 \quad (2)$$

Where, $\sigma_c$ and $k_c$ are casson yield stress (Pa) and casson viscosity ($Pa^{0.5}$ $s^{0.5}$), respectively.

### Statistically analysis

Treatments were triplicated three times and data obtained were analyzed using a one-way analysis of variance (ANOVA) with significance at $p < 0.05$. Significant differences among mean values were determined by using Duncan's multiple range test (Duncan 1955).

## Results and discussion

### Milling and chemical composition

The milled product extraction details and their chemical composition are presented in the Fig. 1. The milling extraction detail shows that FL fraction yield was higher (51.36 %) followed by the CH fraction (39.68 %). Flour fraction comes from the embryo part of the fenugreek seed. In mature seed the embryo weight is higher than the husk weight. The CH is outer portion of seed and consists of testa and endosperm. FH milled fraction observed the yield of 5.23 % and contained damaged husk and embryo. The PL fraction is more of embryo with less husk portion in it.

The moisture content was higher in the CH fraction than other milled fraction, as CH fraction is obtained at early stage of milling. This resulted in less moisture loss during the milling. CH fraction was high in total dietary fibre (TDF) with high soluble dietary fiber (SDF). The SDF content of fenugreek seed is mainly composed of soluble galactomannan gum. The CH part contains endosperm, which swells up in water and forms a thick gelatinous sac. The FL fraction from embryo fraction was high in protein (42.15 %) and in fat (13.63 %) content. The milling yield and chemical composition of the fenugreek fractions are in agreements with the Sakhare et al. 2014b.

### Color measurements

The color measurement data of roller milled fractions of fenugreek is presented in Table 1. The highest brightness value (L) of 82.2 was observed for the flour fraction, which was decreased for pollard (68.8), fine husk (63.54) and coarse husk (60.25) and the lowest yellowness value (+b) of 30.48 was found for coarse husk, whereas flour showed highest yellowness value of 42.15. The embryo part of seed is yellowish in color and contributes to the FL fraction. Coarse and fine husk fractions were observed 6.23 and 4.12 values for the

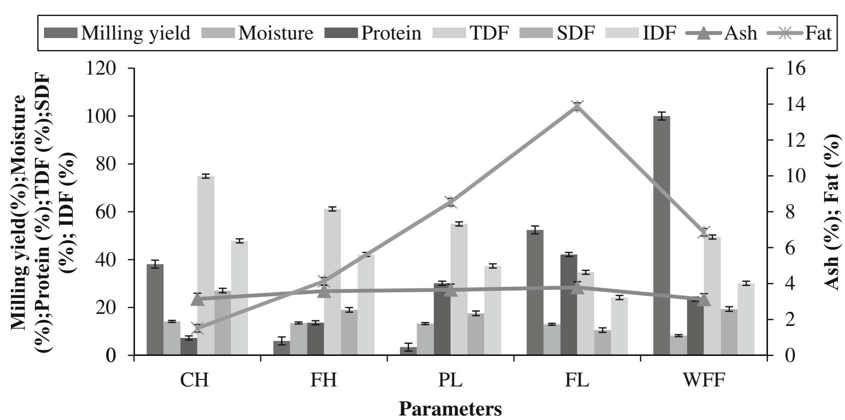


**Fig. 1** Milling yield and chemical composition of roller milled fenugreek fractions WFF: whole fenugreek flour; CH: coarse husk; FH: fine husk; PL: pollard; FL: flour. Error bars indicate the standard deviation of replicates

**Table 1** Color measurement of fenugreek roller milled fractions

| Samples | Color Values | | | |
|---|---|---|---|---|
| | L | a | b | ΔE |
| WFF | 77.55 ± 0.27[a] | 2.29 ± 0.06[d] | 20.95 ± 0.19[c] | 23.59 ± 0.15[b] |
| CH | 62.62 ± 0.43[e] | 4.45 ± 0.17[a] | 16.15 ± 0.21[e] | 31.46 ± 0.48[c] |
| FH | 64.75 ± 0.4[d] | 3.94 ± 0.18[b] | 17.24 ± 0.26[d] | 31.81 ± 0.51[c] |
| PL | 66.89 ± 0.74[c] | 3.04 ± 0.18[c] | 24.03 ± 0.19[b] | 31.82 ± 0.71[c] |
| FL | 73.63 ± 0.35[b] | 1.73 ± 0.13[e] | 31.73 ± 0.06[a] | 33.67 ± 0.28[a] |

Mean values ($n = 3$) ± SD

Values in the column with the same letter in superscript are not significantly different from each other ($p \leq 0.05$)

*WFF* whole fenugreek flour; *CH* coarse husk; *FH* fine husk; *PL* pollard; *FL* flour

redness. The redness value (+a) of flour was 1.34, indicating less contamination of husk fraction.

## Characterization of flow properties

A typical rheograms for the roller milled fractions and WFF paste assessed at 30 °C temperature both in the forward (increasing shear rate) and backward (decreasing shear rate) directions are shown in Fig. 2 for comparison. Higher shear stress was observed for the back ward curves than those of forward curves for all fractions and WFF at all temperatures. The rheograms showed presence of hysteresis loops, which indicates a continual build-up of structure with the shear (over) time. This kind of flow behavior is an indication of presence of strong binding forces which prevent the paste from displacement of particles, and the substance behaves as a solid formed. These results were in accordance with Işıklı and Karababa (2005). All roller milled fractions and WFF exhibited non-Newtonian, shear thinning (pseudoplastic) behaviour. The rheological data obtained were determined using the Power law and Casson models because they are commonly used to describe the rheological behavior of fluids exhibiting no plastic behavior (Steffe 1996).

The parameters obtained by the Casson model (Eq. 1) and power law model (Eq. 2) are summarized in Table 2 and Table 3 respectively. The Casson model gave a relatively poor fit for the experimental data, especially for backward curves with coefficient of determination ($R^2$) values ranging from 0.71 to 0.81, 0.63 to 0.76, 0.61 to 0.78, 050 to 0.66 and 0.79 to 0.91 for WWF, CH, FH, PL and FL respectively (Table 2). Power law model showed better fit for data with coefficient of determination ($R^2$) value generally above 0.94 (Table 3). Among power law parameters, flow behavior index (n) and consistency coefficient (m) were used to compare rheological

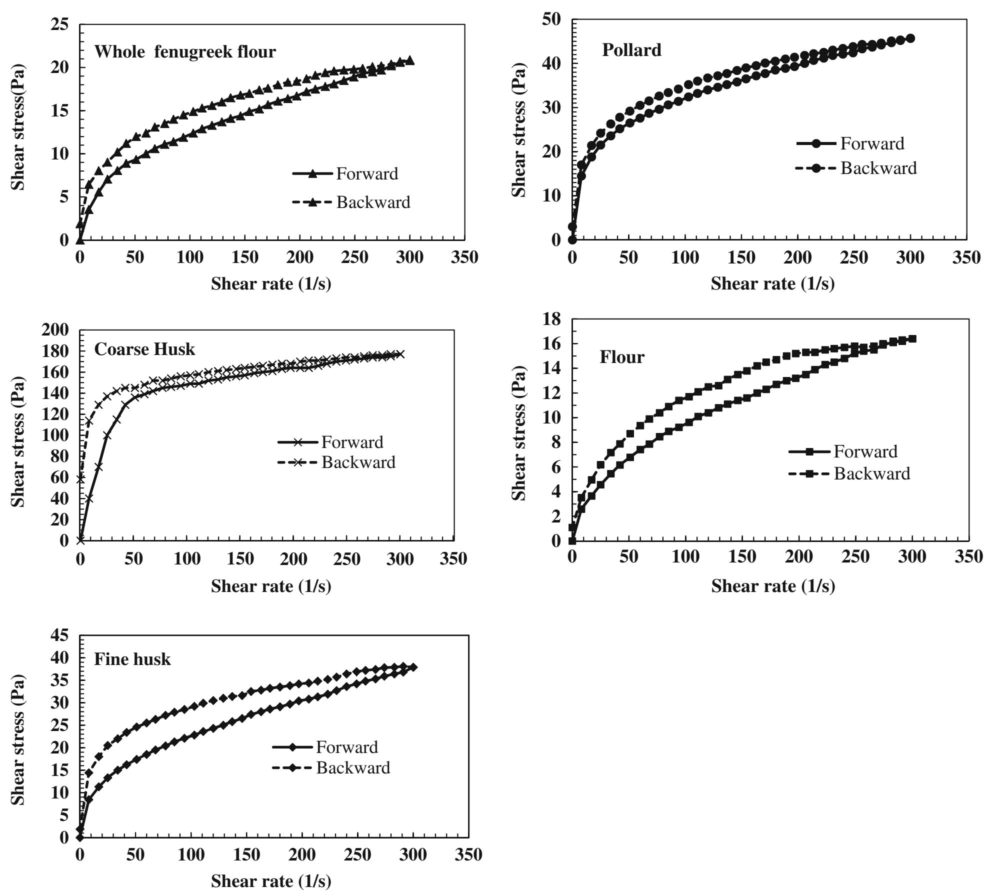


**Fig. 2** Typical flow curve of fenugreek roller milled fractions and WFF at 30 °C

characteristics of the forward and backward curves for all fractions and temperatures. Flow curves obtained for roller milled fractions and WFF pastes together with power law model fits obtained at 30 $^0$C temperatures are presented in Fig. 3.

The results showed that the relationship between shear stress-shear rates is non-linear, indicating that all fractions and WFF behaves non-Newtonian fluid. The values of flow index (n) were less than 1 for all roller milled fraction and WFF at all temperatures suggesting that they are pseudoplastic coating materials. When WFF and ground fenugreek roller milled fractions mixed with water swelling and disruption of granules produce viscous mass, which easily broken and disintegrated by increasing of shear rate. The flow curve data showed higher consistency coefficient and lower flow behavior index for backward curves than those of forward curves. This finding shows that roller milled fractions and WFF paste has a thickening structure. Among the fractions, CH showed 63.25, the highest consistency coefficient followed by PL (7.27), FH (3.40) and FL 1.05) for the forward curve at

**Table 2** Casson model parameters for roller milled fractions of fenugreek and WFF paste at different temperatures

| Sample | Temperatures (°C) | Casson Model | | | | | |
|---|---|---|---|---|---|---|---|
| | | Forward measurement | | | Backward measurement | | |
| | | $\sigma_c$ (Pa) | kc ($Pa^{0.5}$ $s^{0.5}$) | $R^2$ | $\sigma_c$ (Pa) | $k_c$ ($Pa^{0.5}$ $s^{0.5}$) | $R^2$ |
| WWF | 10 | 1.76 ± 0.03 $^d$ | 0.14 ± 0.01 $^e$ | 0.97 | 1.17 ± 0.01 $^e$ | 0.19 ± 0.01 $^d$ | 0.79 |
| | 20 | 1.87 ± 0.02 $^d$ | 0.15 ± 0.00 $^e$ | 0.95 | 1.20 ± 0.02 $^e$ | 0.20 ± 0.00 $^c$ | 0.67 |
| | 30 | 1.93 ± 0.03 $^d$ | 0.15 ± 0.00 $^e$ | 0.97 | 1.27 ± 0.02 $^e$ | 0.21 ± 0.00 $^c$ | 0.81 |
| | 40 | 2.17 ± 0.05 $^d$ | 0.17 ± 0.01 $^c$ | 0.96 | 1.40 ± 0.03 $^e$ | 0.21 ± 0.00 $^d$ | 0.73 |
| | 50 | 2.32 ± 0.04 $^d$ | 0.28 ± 0.01 $^b$ | 0.99 | 1.48 ± 0.03 $^d$ | 0.21 ± 0.01 $^d$ | 0.71 |
| | 60 | 2.58 ± 0.06 $^d$ | 0.30 ± 0.01 $^b$ | 0.98 | 1.75 ± 0.05 $^e$ | 0.25 ± 0.02 $^d$ | 0.72 |
| CH | 10 | 7.63 ± 0.10 $^a$ | 0.15 ± 0.01 $^d$ | 0.96 | 8.44 ± 0.11 $^a$ | 0.18 ± 0.05 $^d$ | 0.63 |
| | 20 | 8.84 ± 0.12 $^a$ | 0.17 ± 0.02 $^d$ | 0.98 | 8.85 ± 0.08 $^a$ | 0.24 ± 0.01 $^b$ | 0.69 |
| | 30 | 9.25 ± 0.05 $^a$ | 0.23 ± 0.04 $^a$ | 0.97 | 9.50 ± 0.02 $^a$ | 0.25 ± 0.01 $^b$ | 0.65 |
| | 40 | 10.17 ± 0.11 $^a$ | 0.19 ± 0.01 $^b$ | 0.90 | 9.15 ± 0.12 $^a$ | 0.28 ± 0.00 $^b$ | 0.59 |
| | 50 | 10.35 ± 0.14 $^a$ | 0.26 ± 0.04 $^c$ | 0.96 | 9.54 ± 0.06 $^a$ | 0.28 ± 0.00 $^b$ | 0.76 |
| | 60 | 10.74 ± 0.15 $^a$ | 0.30 ± 0.05 $^b$ | 0.81 | 9.70 ± 0.10 $^a$ | 0.30 ± 0.01 $^b$ | 0.64 |
| FH | 10 | 1.90 ± 0.11 $^c$ | 0.21 ± 0.00 $^a$ | 0.97 | 1.37 ± 0.15 $^c$ | 0.24 ± 0.00 $^b$ | 0.62 |
| | 20 | 2.16 ± 0.05 $^c$ | 0.21 ± 0.00 $^a$ | 0.99 | 2.19 ± 0.10 $^a$ | 0.24 ± 0.01 $^b$ | 0.78 |
| | 30 | 2.54 ± 0.16 $^c$ | 0.21 ± 0.00 $^b$ | 0.99 | 2.38 ± 0.06 $^b$ | 0.25 ± 0.00 $^b$ | 0.61 |
| | 40 | 2.67 ± 0.06 $^c$ | 0.21 ± 0.01 $^a$ | 0.97 | 2.45 ± 0.02 $^b$ | 0.25 ± 0.00 $^c$ | 0.75 |
| | 50 | 2.80 ± 0.03 $^c$ | 0.31 ± 0.01 $^a$ | 0.91 | 2.47 ± 0.01 $^b$ | 0.26 ± 0.01 $^c$ | 0.73 |
| | 60 | 2.87 ± 0.05 $^c$ | 0.31 ± 0.01 $^a$ | 0.95 | 2.55 ± 0.06 $^b$ | 0.28 ± 0.01 $^c$ | 0.76 |
| PL | 10 | 3.32 ± 0.02 $^b$ | 0.18 ± 0.01 $^c$ | 0.98 | 1.54 ± 0.16 $^b$ | 0.32 ± 0.00 $^a$ | 0.52 |
| | 20 | 3.43 ± 0.06 $^b$ | 0.19 ± 0.00 $^b$ | 0.98 | 1.74 ± 0.03 $^c$ | 0.32 ± 0.01 $^a$ | 0.51 |
| | 30 | 3.52 ± 0.05 $^b$ | 0.19 ± 0.01 $^c$ | 0.97 | 1.78 ± 0.04 $^c$ | 0.33 ± 0.00 $^a$ | 0.50 |
| | 40 | 3.57 ± 0.02 $^b$ | 0.19 ± 0.01 $^b$ | 0.97 | 1.92 ± 0.15 $^c$ | 0.34 ± 0.00 $^a$ | 0.54 |
| | 50 | 3.63 ± 0.02 $^b$ | 0.19 ± 0.00 $^d$ | 0.97 | 2.12 ± 0.09 $^c$ | 0.34 ± 0.01 $^a$ | 0.66 |
| | 60 | 3.66 ± 0.02 $^b$ | 0.20 ± 0.01 $^c$ | 0.97 | 2.28 ± 0.11 $^c$ | 0.34 ± 0.01 $^a$ | 0.56 |
| FL | 10 | 1.63 ± 0.08 $^e$ | 0.19 ± 0.01 $^b$ | 0.96 | 1.29 ± 0.04 $^d$ | 0.23 ± 0.01 $^b$ | 0.88 |
| | 20 | 1.52 ± 0.10 $^e$ | 0.18 ± 0.01 $^c$ | 0.97 | 1.14 ± 0.03 $^d$ | 0.21 ± 0.01 $^c$ | 0.81 |
| | 30 | 1.47 ± 0.02 $^e$ | 0.17 ± 0.00 $^d$ | 0.98 | 1.10 ± 0.04 $^d$ | 0.19 ± 0.00 $^d$ | 0.79 |
| | 40 | 1.29 ± 0.05 $^e$ | 0.17 ± 0.01 $^c$ | 0.96 | 1.05 ± 0.02 $^d$ | 0.19 ± 0.01 $^e$ | 0.84 |
| | 50 | 1.21 ± 0.03 $^e$ | 0.15 ± 0.00 $^e$ | 0.98 | 0.98 ± 0.03 $^e$ | 0.18 ± 0.00 $^e$ | 0.86 |
| | 60 | 1.12 ± 0.04 $^e$ | 0.14 ± 0.01 $^d$ | 0.98 | 0.91 ± 0.00 $^d$ | 0.17 ± 0.01 $^e$ | 0.91 |

$R^2$ is determination coefficient

*WWF* Whole fenugreek flour; *CH* Coarse husk; *FH* Fine husk; *PL* Pollard; *FL* Flour

Mean values ($n = 3$) ± SD.Values in same column and for the same temperature with the same letter in superscript are not significantly different from each other ($p \leq 0.05$)

30 $^0$C temperatures. This indicates that the CH forms the more viscous paste among the roller milled fractions and has a potential to be used as a thickening agent. This is in agreement with the chemical composition of CH, which showed higher SDF content. The fenugreeks SDF are mainly the galactomannans which have a property of high water-binding capacity and forms of highly viscous solutions at relatively low concentrations. The consistency coefficient (m) increased from 34.84 to 82.44 and 58.87 to 86.26 for CH fraction for forward and backward curve respectively. The consistency coefficient (m), increased with increasing temperatures for CH, FH, PL and WFF. This behavior showed that when roller milled fractions (CH, FH, PL) and WFF water suspension is heated, granules absorbs water, forcing them swell and forms a highly viscous paste. In general the apparent viscosity of a liquid decreases with increase in temperature, but in the present investigation, apparent viscosity and consistency coefficient increased with increasing temperature for CH, FH, PL and WFF except for FL fraction. This behavior could be due to an increase in water solubility of higher molecular mass compounds with temperature and also change in the soluble mass ratio of galactose to mannose in the aqueous suspension. Our results

**Table 3** Power law parameters for roller milled fractions of fenugreek and WFF paste at different temperatures

| | | Power law model | | | | | |
|---|---|---|---|---|---|---|---|
| Sample | Temperature (°C) | Forward measurement | | | Backward measurement | | |
| | | *m* (Pas$^n$) | *n* | $R^2$ | *m* (Pas$^n$) | *n* | $R^2$ |
| WFF | 10 | 0.48 ± 0.54 $^e$ | 0.55 ± 0.02 $^a$ | 0.99 | 3.13 ± 0.06 $^e$ | 0.31 ± 0.00 $^c$ | 0.96 |
| | 20 | 1.21 ± 0.82 $^d$ | 0.47 ± 0.01 $^b$ | 0.99 | 3.36 ± 0.04 $^e$ | 0.30 ± 0.02 $^c$ | 0.98 |
| | 30 | 1.87 ± 0.23 $^d$ | 0.40 ± 0.01 $^c$ | 0.99 | 3.50 ± 0.08 $^d$ | 0.29 ± 0.01 $^b$ | 0.96 |
| | 40 | 1.99 ± 0.71 $^d$ | 0.39 ± 0.00 $^b$ | 0.99 | 3.90 ± 0.12 $^d$ | 0.29 ± 0.00 $^c$ | 0.99 |
| | 50 | 2.06 ± 0.49 $^d$ | 0.39 ± 0.00 $^b$ | 0.99 | 4.47 ± 0.09 $^d$ | 0.26 ± 0.01 $^c$ | 0.99 |
| | 60 | 2.52 ± 0.55 $^d$ | 0.38 ± 0.01 $^b$ | 0.98 | 4.91 ± 0.06 $^d$ | 0.24 ± 0.01 $^d$ | 0.97 |
| CH | 10 | 35.84 ± 1.12 $^a$ | 0.26 ± 0.01 $^e$ | 0.99 | 58.87 ± 1.23 $^a$ | 0.12 ± 0.02 $^e$ | 0.93 |
| | 20 | 51.70 ± 1.62 $^a$ | 0.21 ± 0.01 $^d$ | 0.99 | 63.38 ± 1.53 $^a$ | 0.15 ± 0.01 $^d$ | 0.93 |
| | 30 | 63.25 ± 0.85 $^a$ | 0.24 ± 0.01 $^e$ | 0.99 | 68.14 ± 2.53 $^a$ | 0.16 ± 0.00 $^d$ | 0.92 |
| | 40 | 74.04 ± 0.61 $^a$ | 0.15 ± 0.02 $^d$ | 0.99 | 76.03 ± 3.11 $^a$ | 0.17 ± 0.01 $^e$ | 0.92 |
| | 50 | 80.21 ± 1.27 $^a$ | 0.18 ± 0.02 $^d$ | 0.98 | 83.32 ± 1.71 $^a$ | 0.18 ± 0.00 $^d$ | 0.94 |
| | 60 | 82.44 ± 0.71 $^a$ | 0.15 ± 0.01 $^d$ | 0.99 | 86.26 ± 0.89 $^a$ | 0.19 ± 0.01 $^e$ | 0.93 |
| FH | 10 | 1.85 ± 0.08 $^c$ | 0.48 ± 0.02 $^b$ | 0.99 | 5.00 ± 0.07 $^c$ | 0.36 ± 0.02 $^a$ | 0.94 |
| | 20 | 2.42 ± 0.07 $^c$ | 0.45 ± 0.02 $^b$ | 0.99 | 5.25 ± 0.10 $^c$ | 0.35 ± 0.00 $^a$ | 0.97 |
| | 30 | 3.40 ± 0.08 $^c$ | 0.42 ± 0.00 $^b$ | 0.99 | 5.41 ± 0.06 $^c$ | 0.35 ± 0.01 $^a$ | 0.96 |
| | 40 | 3.79 ± 0.05 $^c$ | 0.40 ± 0.01 $^b$ | 0.99 | 5.86 ± 0.08 $^c$ | 0.34 ± 0.00 $^b$ | 0.94 |
| | 50 | 4.23 ± 0.07 $^c$ | 0.39 ± 0.00 $^b$ | 0.98 | 6.08 ± 0.11 $^b$ | 0.34 ± 0.00 $^b$ | 0.95 |
| | 60 | 4.46 ± 0.11 $^c$ | 0.38 ± 0.01 $^b$ | 0.99 | 6.29 ± 0.08 $^b$ | 0.33 ± 0.01 $^b$ | 0.95 |
| PL | 10 | 6.15 ± 0.22 $^b$ | 0.33 ± 0.00 $^d$ | 0.99 | 7.67 ± 0.15 $^b$ | 0.33 ± 0.01 $^b$ | 0.95 |
| | 20 | 6.63 ± 0.08 $^b$ | 0.33 ± 0.00 $^c$ | 0.99 | 7.12 ± 0.21 $^b$ | 0.34 ± 0.00 $^b$ | 0.95 |
| | 30 | 7.27 ± 0.12 $^b$ | 0.32 ± 0.01 $^d$ | 0.99 | 6.65 ± 0.07 $^b$ | 0.35 ± 0.01 $^a$ | 0.94 |
| | 40 | 7.59 ± 0.14 $^b$ | 0.32 ± 0.00 $^c$ | 0.99 | 6.20 ± 0.08 $^b$ | 0.36 ± 0.00 $^a$ | 0.94 |
| | 50 | 7.91 ± 0.06 $^b$ | 0.31 ± 0.00 $^c$ | 0.99 | 5.99 ± 0.10 $^c$ | 0.36 ± 0.01 $^a$ | 0.94 |
| | 60 | 8.22 ± 0.10 $^b$ | 0.31 ± 0.00 $^c$ | 0.99 | 5.44 ± 0.16 $^c$ | 0.37 ± 0.00 $^a$ | 0.95 |
| FL | 10 | 1.47 ± 0.11 $^d$ | 0.45 ± 0.01 $^c$ | 0.99 | 3.63 ± 0.12 $^d$ | 0.30 ± 0.00 $^d$ | 0.96 |
| | 20 | 1.12 ± 0.08 $^e$ | 0.48 ± 0.01 $^a$ | 1.00 | 3.39 ± 0.10 $^d$ | 0.29 ± 0.01$^c$ | 0.93 |
| | 30 | 0.92 ± 0.05 $^e$ | 0.49 ± 0.00 $^a$ | 1.00 | 3.10 ± 0.08 $^e$ | 0.26 ± 0.01 $^c$ | 0.94 |
| | 40 | 0.77 ± 0.06 $^e$ | 0.50 ± 0.00 $^a$ | 1.00 | 2.68 ± 0.24 $^e$ | 0.27 ± 0.00 $^d$ | 0.94 |
| | 50 | 0.55 ± 0.10 $^e$ | 0.52 ± 0.01 $^a$ | 0.99 | 2.38 ± 0.14 $^e$ | 0.27 ± 0.00 $^c$ | 0.96 |
| | 60 | 0.31 ± 0.07 $^e$ | 0.50 ± 0.01 $^a$ | 1.00 | 2.01 ± 0.09 $^e$ | 0.26 ± 0.01 $^c$ | 0.94 |

Power-law parameters: *m* - consistency coefficient; n - flow behavior index; $R^2$ - determination coefficient

*WWF* Whole fenugreek flour; *CH* Coarse husk; *FH* Fine husk; *PL* Pollard; *FL* Flour

Mean values ($n = 3$) ± SD.Values in same column and for the same temperature with the same letter in superscript are not significantly different from each other ($p \leq 0.05$)

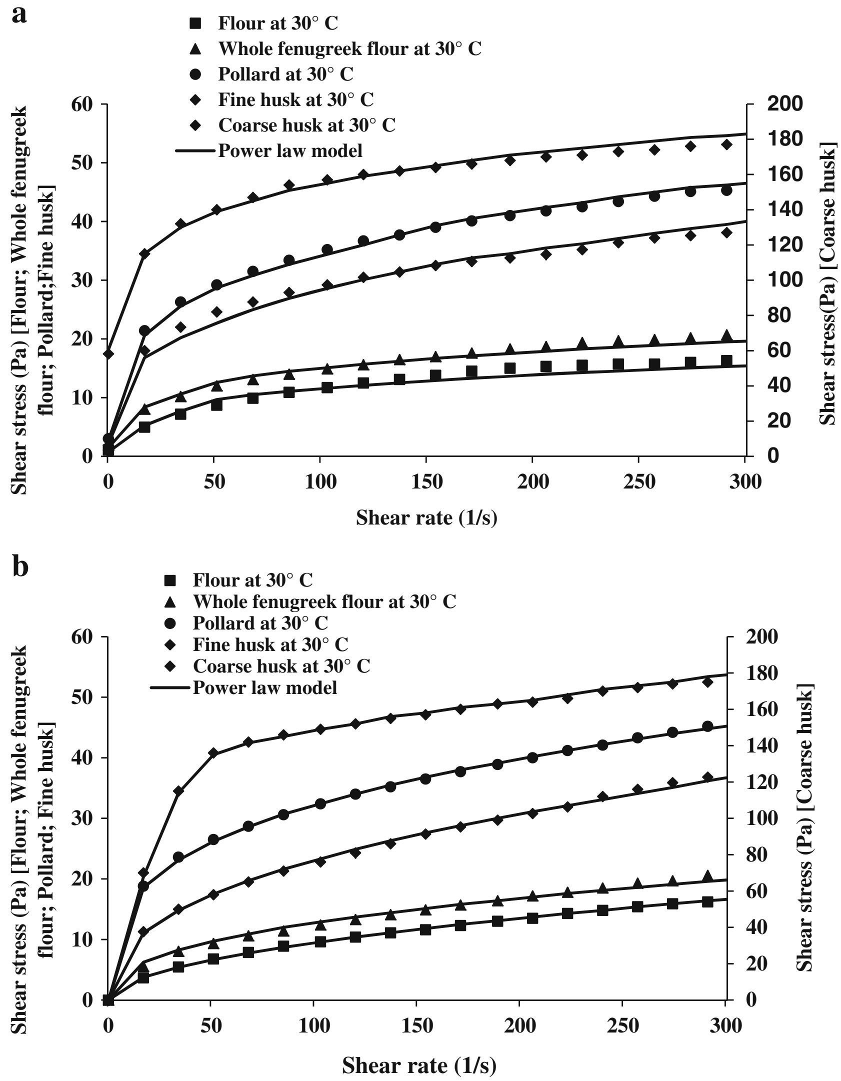


**Fig. 3** Flow curve of fenugreek roller milled fraction and whole fenugreek paste fitted to power law model at 30 °C. **a** Forward measurement; **b** Backward measurement

are in agreements with the observation of Işıklı and Karababa (2005), which showed that apparent viscosity increased with increase in temperature. Garcia-Ochoa and Casas (1992) in their locust bean gum (LBG) showed that when the solubilisation temperature increases, an increase in apparent viscosity is observed. The LBG contain the high concentration of galactomannan. They explained that, this behavior of apparent viscosity of aqueous suspension of LBG is due to the solubilisation of some compounds in gum. The water solubility of some compound in gum increases and the mass ratio of galactose to mannose decreases with increase of temperature up to 80 $^0$C. This resulted in increase in viscosity of aqueous suspension. Earlier researchers also observed that the temperature dependent viscosity must be due to differences in the number of inter and/or intramolecular bonds of the compounds of the gums. Casas and Garcia-Ochoa (1999) also reported an increase in viscosity with increased temperature up to 60 $^0$C for the solution of xanthan and locust bean gum mixtures. For FL fractions, consistency coefficient (m), decreased as the temperatures increased.

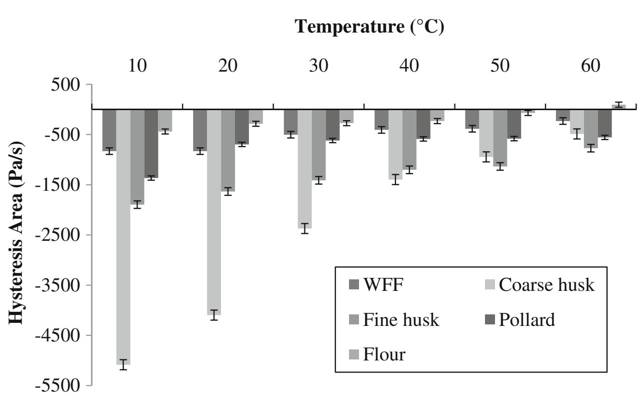


**Fig. 4** Hysteresis and relative hysteresis area of fenugreek roller milled fractions and WFF pates

Hysteresis loop area for all roller milled fractions and WFF paste assessed at different temperature are shown in Fig. 4. The type of fenugreek fractions, its composition and temperature significantly affected the hysteresis area. All fenugreek fractions and WFF showed negative hysteresis area, except FL fraction at 60 $^0$C. Hysteresis area was large for CH and decreased for the FH, PL and FL fractions for given temperature.

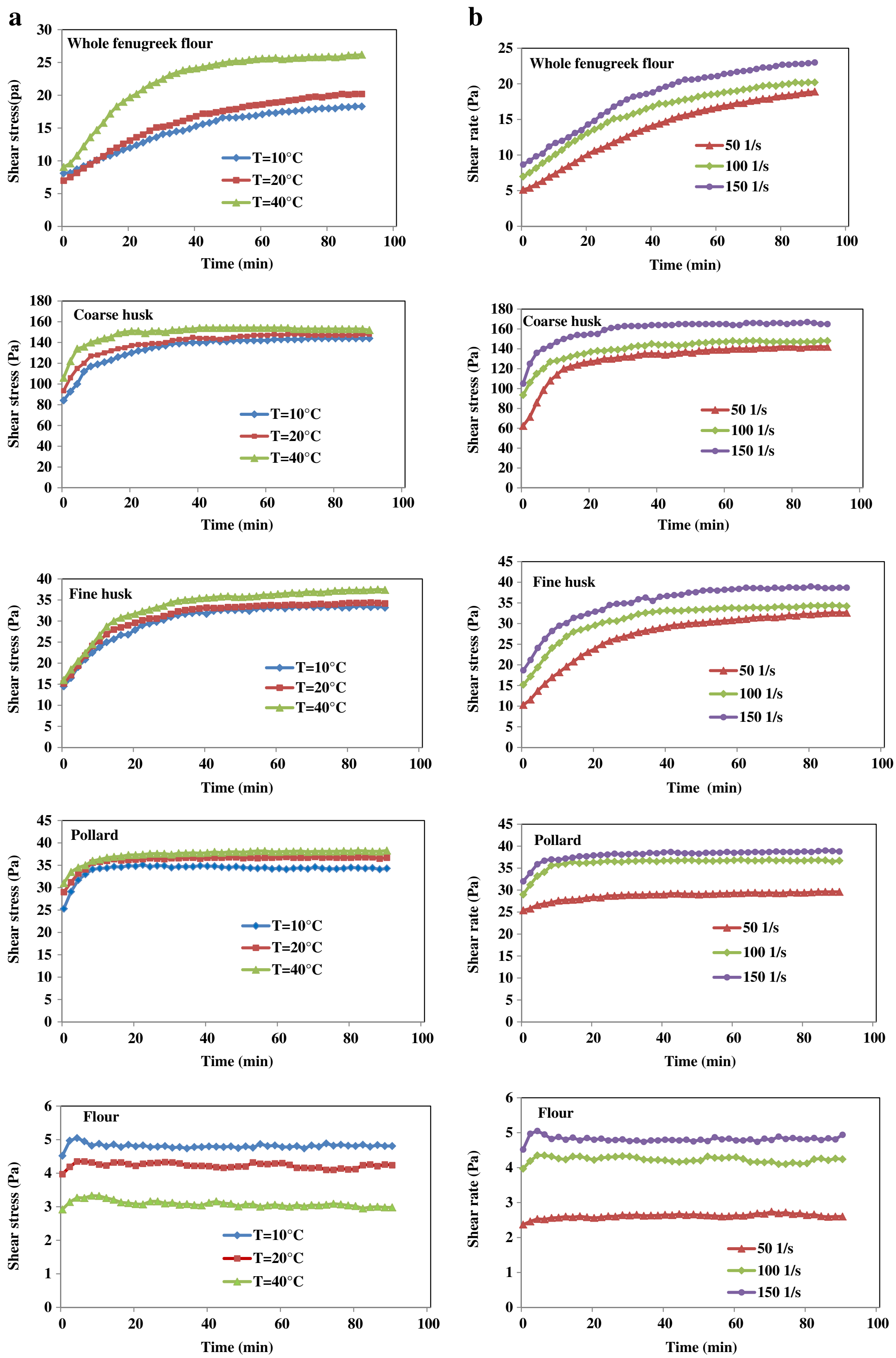


**Fig. 5** A: Effect of temperature on stress of fenugreek roller milled fractions and whole fenugreek flour paste at constant shear speed. B: Effect of shear speed on stress of fenugreek roller milled fractions and whole fenugreek flour paste at constant temperature

The hysteresis area observed for the CH at 10 $^0$C was 6.12, 2.68, 3.72 and 11.57 times higher than WFF, FH, PL and FL fractions at same temperature. This could be related to the SDF content of samples, which was high in CH followed by FH, PL, WFF and FL. Hysteresis area decreased with increase in temperatures as high as 10 $^0$C and low at 60 $^0$C for all the samples. This could be probably because of effect of temperature on molecular structure damage. At lower temperature hysteresis area was significantly smaller than at higher temperature for all the samples. At same temperature, hysteresis loop showed different form and magnitude, which was noticeable at low temperature. The area obtained at 60 $^0$C for was less 42 % than the values at 10 $^0$C for the FH, PL and WFF. The CH observed 90 % less area at 60 °C compared to values at 10 $^0$C (Fig. 4).

### Time-dependent flow properties

Time dependent flow properties of fenugreek roller milled fractions and WFF were measured by shearing the sample at constant shear rate of 100 $s^{-1}$ at different temperatures and data obtained are shown in Fig. 5A. The shear stress increased with increase in temperature at constant shear rate for CH, FH, PL and WFF where as decreased for FL fraction with increase in temperature. Increase in the shear stress of samples with increase of time can be caused by rheopectic behavior. This behavior could be due to an increase in water solubility of some compounds in fenugreek powder. The fenugreek fractions and WFF paste became a viscous material by increased rate of swelling and hydration of fenugreek particles with time of shearing. We also observed that equilibrium time decreased with increase in temperature. The shear stress increased rapidly with time within first 40 min and approached equilibrium state after approximately 70 min for WFF paste at constant shear rate. In case of CH and PL shear stress increased within 10 to 20 min and afterwards approached to equilibrium state. In case of FL, shear stress increased first 3 min and decreased afterwards. Among all roller milled fractions, CH observed higher shear stress followed by Pl, FH and SRF at given time. At 20 $^0$C temperature and 20 min time the CH, PL, FH, WFF and FL showed 137, 36.3, 29.6, 13.1 and 4.22 Pa shear stress respectively.

The effects of different shear rate on the shear stress are shown in Fig. 5B. The shear stress increased with increase in shear rate at constant temperature. The value of shear stress at different shear rate increased for 10–15 min and reached to the equilibrium value at approximately 20 min for the CH and PL. In case of WFF, shear stress increased rapidly for 60 min and approached constant value at about 80 min. The roller milled fraction showed shear tress value of 155, 37.9, 35.9, 14.3 and 4.8 Pa for CH, PL, FH, WFF and FL at 150 $s^{-1}$ shear rate at 20 min shear time respectively.

## Conclusion

The roller milled fractions and WFF paste exhibited non-Newtonian shear thinning and antitixotropic behaviour for all temperature tested (10-60 °C). The data of forward and backward flow curves showed the flow behavior index, n less than unity for all the studied samples at all temperature tested. The consistency coefficient (m) increased as the temperature increases for CH, FH, PL and WFF paste and decreased with increased temperature for the flour fractions paste. The value for consistency coefficient was higher for the CH among all fractions. The hysteresis loop area was large for the CH and small for FL implying more and less damage to the structures respectively. All roller milled fractions and WFF pastes became less rheopectic at higher temperature. Time dependent flow properties showed high shear stress with increase in shear rate at constant temperature and increase in temperature at constant shear rate.

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