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# Steady and oscillatory shear behaviour of semi-concentrated starch suspensions

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**Abstract**

The viscoelastic moduli G' and G" of aqueous suspensions with 40% (w/v) normal corn starch (NCS) and waxy corn starch (WCS) were determined by oscillation rheometry. The oscillatory shear flow experiments at heating from 30° to 75°C and maintaining at this temperature showed changes from a behaviour predominant viscous (G">>G') to predominant elastic (G'>G") for both starches at 60.5°C for WCS, respectively 70,85°C for NCS, WCS having higher values of G' and G" as NCS. After the gelatinisation temperature was attired, NCS showed no significant changes, both moduli remaining relatively constant. Peaks of both moduli G' and G" were obtained for WCS at its maintaining at 75°C, these changes being attributed to the changes in the amylopectin structure in the absence of amylose for this starch type. The frequency influenced the results; analysis at constant low frequency ($10s^{-1}$) gave big oscillations during the measurements and made the analysis impossible, whereas frequencies as $50s^{-1}$ or $100s^{-1}$ gaves reproducible and similar results. The shear flow measurements realised at angular frequencies ω from$10^{-1}$ to $10^{3}s^{-1}$ at 25°C showed that changes from a behaviour predominant elastic (G'>G") to predominant viscous (G">>G') occurred when ω attired the values $10s^{-1}$ for WCS and $3s^{-1}$ for NCS. The calculation of the 'Power-Law' parameter B showed that NCS forms a physical gel structure, whereas WCS behaves as a covalent gel in the frequency domain $10^{-1}$ to 10 $s^{-1}$ and as physical gel in the frequency domain 10 to $10^{2}$ $s^{-1}$.



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*Keywords*: corn starch;waxy corn starch; steady shear; oscillatory shear; viscoelastic moduli

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## 1. Introduction

Corn starch is a valuable raw material at the obtaining of starch hydrolysates as maltodextrins, glucose or maltose syrups by using enzymes, acid or by combined acid-enzymatic hydrolysis [1]. The enzymatic hydrolysis of starch occurs in three steps: gelatinisation, liquefaction and saccharification.

Starch gelatinisation is commonly defined as an irreversible order–disorder transition of starch granules from a structured state to a disordered state (starch pastes) upon heating [2]. Upon heating of a starch suspension, the granules swell due to absorption of moisture in the amorphous regions of the granule and amylose (AMY) and water start diffusing out of the granular entities [3]. At higher temperatures, amylopectin (AMP) melting begins and enables the release of the important amount of AMY that was trapped between AMP layers and of the small part that was confined within crystalline amylopectin; the crystalline order of AMP losses and so the birefringence [3][4]. Finally, a gel or a paste-like mass is formed [5]. The structure of gel or paste is affected by the starch concentration and the structure of swollen starch granules, the amounts and the types of AMY and AMP leached out, and heating conditions such as temperature, heating time and heating rate [6].

Starch exhibits unique rheological behaviour with change of temperature, concentration and shear rate., rheology being a valuable tool to predict the product properties and select the raw materials for food production [7]. Rheological behaviour of concentrated starch suspensions was reported to be affected by AMY [8]. Amylose molecules begin to leach from the granules and the viscosity increases, maximal viscosity corresponding to the point when the number of swollen but still intact starch granules is maximal. The maximum is followed by a decrease in paste viscosity, as the granules rupture and starch molecules are dispersed in the aqueous phase. The rate and extent of breakdown are dependent on the type and amount of starch, the temperature gradient, shear force and the composition of the mixture [8].

The gelatinization temperature of most starches is between 60 and 80oC. In general, there is a negative relationship between the AMY content of starch and the gelatinization temperature and peak viscosity [9].

This research investigated the dynamic rheological behaviour of concentrated corn starch suspension by small amplitude oscillatory shear tests and steady shear tests. Waxy corn starch consist of essentially 100% AMP, effects of leached-out AMY on the gelation of starch dispersions are negligible. Therefore, two starch types: normal corn starch (NCS) and waxy corn starch (WCS) are used in this study for elucidating the effect of composition on the change of viscoelastic properties during heating at temperatures corresponding to the gelatinisation.

## 2. Materials and Methods

Two types of corn starch were used: native corn starch (NCS) and waxy corn starch (WCS), with 78% AMP and 13.18 μm granule mean diameter, respectively with 99% AMP and 9.5 μm granule mean diameter. 40% (w/v) aqueous starch solutions were prepared by stirring at 25$^{o}$C.

Small amplitude oscillatory rheological measurements were performed using a rotational rheometer Rotovisco RV100 (Haake, Germany) with temperature controlling system TCP/P and cone-plate geometry (C60/1). Two types of measurements were done: oscillatory shear flow and steady shear flow.

In the oscillatory shear flow experiments the sample is exposed to a forced oscillation and the transmitted stress is measured. Derived parameters include storage and loss moduli (G' and G"), where the storage parameter is related to elastic energy and the loss parameter to viscous flow. For the analysis of starch suspensions in the oscillatory mode, measurements at three constant frequencies ω (10, 50 and 100 $s^{-1}$) were made. The heating rate was linear from 30° to 75°C with heating rate 0.18°C/sec.

For structural fluids, such as starch suspensions or starch gels, small amplitude oscillatory shear flow may be the only way to find their rheological characteristics. By working with suspensions, small amplitude tests are recommended [10], because of the possibility of structure breakdown at large shear