



In situ rheometry of concentrated cellulose fibre suspensions and relationships with enzymatic hydrolysis



Tien-Cuong Nguyen^{a,*}, Dominique Anne-Archard^b, Véronique Coma^c, Xavier Cameleyre^a, Eric Lombard^a, Cédric Binet^b, Arthur Nouhen^b, Kim Anh To^d, Luc Fillaudeau^a

^a Laboratoire d'Ingénierie des Systèmes Biologiques et des Procédés (Université de Toulouse, INSA, INRA UMR792, CNRS UMR5504), Toulouse, France

^b Université de Toulouse, INPT, UPS, IMFT (Institut de Mécanique des Fluides de Toulouse), Toulouse, France

^c Laboratoire de Chimie des Polymères Organiques UMR 5629 CNRS/Université Bordeaux 1, IPB/ENSCP, Pessac, France

^d School of Biotechnology and Food Technology, Hanoi University of Sciences and Technology, Viet Nam

HIGHLIGHTS

- ▶ We explore the suspending and enzymatic hydrolysis of microcrystalline cellulose, Whatman paper and extruded paper-pulp.
- ▶ A methodology to determine on-line viscosity is proposed and validated.
- ▶ A structured rheological model is established.
- ▶ Suspension viscosity and particle size decreased rapidly during the enzymatic hydrolysis.

ARTICLE INFO

Article history:

Received 15 November 2012

Received in revised form 18 January 2013

Accepted 19 January 2013

Available online 8 February 2013

Keywords:

Lignocellulose

Rheology

Paper pulp

Hydrolyse

Viscosity

ABSTRACT

This work combines physical and biochemical analyses to scrutinize liquefaction and saccharification of complex lignocellulose materials. A multilevel analysis (macroscopic: rheology, microscopic: particle size and morphology and molecular: sugar product) was conducted at the lab-scale with three matrices: microcrystalline cellulose (MCC), Whatman paper (WP) and extruded paper-pulp (PP). A methodology to determine on-line viscosity is proposed and validated using the concept of Metzner and Otto (1957) and Rieger and Novak's (1973). The substrate suspensions exhibited a shear-thinning behaviour with respect to the power law. A structured rheological model was established to account for the suspension viscosity as a function of shear rate and substrate concentration. The critical volume fractions indicate the transition between diluted, semi-diluted and concentrated regimes. The enzymatic hydrolysis was performed with various solid contents: MCC 273.6 gdm/L, WP 56.0 gdm/L, PP 35.1 gdm/L. During hydrolysis, the suspension viscosity decreased rapidly. The fibre diameter decreased two fold within 2 h of starting hydrolysis whereas limited bioconversion was obtained (10–15%).

Crown Copyright © 2013 Published by Elsevier Ltd. All rights reserved.

1. Introduction

Lignocellulose biomass is one of the most abundant renewable resources and certainly one of the least expensive. Its conversion into ethanol fuel is eventually expected to provide a significant portion of the world's energy requirements. The substrates used

are varied. They include woody substrates (hardwood and softwood), products from agriculture (straw) or those of lignocellulosic waste industries (food processing, paper).

In order to achieve economic viability, the biorefining of lignocellulosic resources must be operated at very high feedstock dry matter content. Paper pulp is quite appropriate for modern biorefining, because it displays a low lignin content, it is free of inhibitory compounds that can perturb fermentations and devoid of microbial contaminants.

Nevertheless, the enzyme liquefaction and saccharification of paper-like pulps are subject to the same constraints as other pulps obtained via alternative methods such as steam explosion or dilute acid hydrolysis. Therefore, a better scientific understanding and, ultimately, good technical control of these critical biocatalytic

Abbreviations: N, Mixing rate (rpm); d, Impeller diameter (m); C, Torque (N.m); P, Power (W); ρ , Density (kg/m^3); Np, Power number; Re, Reynolds number; Re_g , Generalized Reynolds number; Re^* , Rieger & Novak Reynolds; μ , Viscosity (Pa.s); $[\mu]$, Intrinsic viscosity; Kp, Geometrical constant; Ks, Metzner-Otto constant; $\dot{\gamma}$, Shear rate (s^{-1}); n, Power-law index; k, Consistency index ($\text{Pa}\cdot\text{s}^n$); Φ , Volume fraction; D[4,3], Mean diameter (μm); C_m , Mass concentration (g/L); dm, Dry matter (g).

* Corresponding author. Tel.: +33 661970369.

E-mail address: tcnguyen@insa-toulouse.fr (T.-C. Nguyen).

reactions, which involve complex matrices at high solid contents, is currently a major challenge if biorefining operations are to become commonplace.

Amongst the main parameters to be studied, the rheological behaviour of the hydrolysis suspension and the fibre particle size of, stand out as a major determinants of process efficiency and determine equipment to be used and the strategies applied (Wiman et al., 2010). The choice of agitation system, fundamental to heat and/or mass transfer, and to disruption of agglomerated particles, influences the bioconversion of cellulose into simple sugar (Um, 2007). It requires detailed knowledge of the rheological behaviour of the substrate suspensions. However, these suspensions present such complex and unique properties that there are no standard method for studying fibre network deformation and pulp flow behaviour (Blanco et al., 2006).

Fibre suspension flow is a key factor and extensive studies have been reported in the pulp and paper scientific literature. Cellulose fibres in suspension form three-dimensional networks that exhibit viscoelastic properties (Wahren et al., 1964; Kerekes et al., 1985 cited by Antunes, 2009). Measuring the rheological properties of fibre suspensions is complex, owing to multiple factors: (i) fibre physical and mechanical properties and concentration ranges, (ii) fibre contacts and surface forces and (iii) forces on fibres and flocculation. Rheological behaviour of fibre suspensions is usually described by an apparent yield stress, a shear viscosity (Herschel–Buckley or Bingham models) and elasticity. The physical properties of cellulose fibre are considered such as swelling, dissolution, structure and strength of network. The strength of the network of the coarsest fibres determines the rheology of these materials (Wiman et al., 2010). The rheology of lignocellulose suspensions is of special interest and studies are numerous at different temperatures and concentrations, from dilute solutions 0.2–3.0% (Agoda-Tandjawa et al., 2010; Ferreira et al., 2003) to concentrated solutions 10–20% (Um and Hanley, 2008; Zhang et al., 2009). Both of these studies conclude that a shear-thinning behaviour occurs for any lignocellulosic substrate suspension: microcrystalline cellulose (Agoda-Tandjawa et al., 2010; Chaussy et al., 2011; Tatsumi and Matsumoto, 2007; Um and Hanley, 2008); hardwood paper-pulp (Blanco et al., 2006; Zhang et al., 2009); softwood paper-pulp (Ferreira et al., 2003; Wiman et al., 2010); sugar cane bagasse (Pereira et al., 2011). The viscosity of the suspension depends not only on the temperature and concentration (Ferreira et al., 2003) but also on the average fibre length (Lapierre et al., 2006). A longer fibre has a higher degree of polymerisation and generates a higher viscosity. During biological hydrolysis, the apparent viscosity of suspensions decreases (Pereira et al., 2011; Um, 2007) in parallel with a decrease of particle size (Wiman et al., 2010).

Traditionally, rotating viscometers have been used (Duffy and Titchener, 1975; Chase et al., 1989; Bennington et al., 1990). However, normal commercial viscometers do not provide enough mixing to maintain uniform fibre distribution, which causes viscosity values close to the viscosity of the pure water (Blanco et al., 1995 cited by Antunes, 2009). Therefore, to study the rheological properties of fibre suspensions there is no standardized method but several measuring devices have been reported in the literature (Cui and Grace, 2007; Blanco et al., 2006; Chaussy et al., 2011; Derakhshandeh et al., 2011). Plate torque-based devices have the highest resolution and can be used to determine the rheological behaviour of pulp suspensions (Blanco et al., 2006). One difficulty remains in the definition of criteria to ascribe a viscosity to a heterogeneous suspension, originally defined for homogeneous fluids in laminar flow (Blanco et al., 2006). To attain fluidisation, apparent yield stress must be exceeded throughout the suspension. Although fluidisation generally occurs in a turbulent regime, fluid-like behaviour at the floc level can be attained under non-turbulent conditions. One example is the flow induced in a rotary

device at slow rotational speeds just above the apparent yield stress; another example was found in spouted beds (Derakhshandeh et al., 2011).

Then on-line measurement of torque or mixing power in bioreactors may highlight viscosity of concentrated cellulose suspensions and may constitute a way to follow enzymatic hydrolysis reactions. Particle size, rheology, and rate of enzymatic hydrolysis could be correlated to operating conditions for example: mixing rate and impeller speed (Pereira et al., 2011; Samaniuk et al., 2011).

The aim of the present report was to investigate the dynamics of transfer phenomena and limitation of biocatalytic reactions with lignocelluloses resources under high concentration conditions. This study focuses on the characterisation of cellulose suspensions at different concentrations and coupling with the enzymatic kinetics of hydrolysis using on-line viscosimetry. In the literature, rheometers are used to determine ex situ suspension viscosity. These approaches are limited by the number of samples and the substrate properties, predominately decantation and flocculation of material. To solve these problems, a method allowing the suspension viscosity to be followed is proposed. Firstly, cellulose fibre suspensions at various concentrations are investigated through on-line measurements in purpose-built bioreactor. Three real and model matrices are characterised by fiber morphology, diameter and concentration. Using Metzner and Otto concept (1957), rheograms were determined. Rheological behaviour was then described by structured rheological models. Secondly, the complex relationships between fibre structure, degradation, chemical composition and rheological behaviour was scrutinised. To do so, physical and biochemical on-line and off-line analyses were conducted during the bioreaction. A relationship between viscosity change and biocatalytic degradation of fibre was observed.

2. Methods

2.1. Experimental device

The experimental set-up consists of a tank and an impeller system connected to a viscometer working at imposed speed (Viscotester HaakeVT550, Thermo Fisher Scientific, Ref: 002-7026) (Fig. 1). This allows on-line torque measurements. The rotational speed ranged between 0.5 and 800 rpm and torque between 1 and 30 mN m. The bioreactor was a homemade glass tank with a flat bottom (diameter: 82 mm, H_{\max} : 76 mm, V: 0.4 L) fitted with a water jacket. The impeller was a four-pitched blade turbine (IKA A200, stainless steel, d : 50 mm, l : 21 mm, w : 8 mm, 45° angle 25 mm from the bottom of the tank to maintain axial and radial flows. Temperature was controlled by circulation (cryostat Haake DC30 and K20) through the water jacket. A bioreactor panel control (B. Braun Biotech International MCU200 + microDCU300) was used for pH control and regulation, dissolved oxygen and temperature measurements. The viscometer and the cryostat were controlled by software from HaakeRheoWin Job Manager (Thermo Fisher Scientific) which also ensured data recording (temperature, torque and mixing rate).

2.2. Substrates and enzymes

Three cellulose matrices were studied in order to investigate different fibre morphologies and particle size distributions (Table 1): microcrystalline cellulose (ACROS Organics, Ref: 382310010), a dried and milled (Bosch MKM6003 mill) Whatman paper (Whatman International Ltd., Maidstone, England, Cat No. 1001 090) and paper-pulp (Tembec Co., Saint-Gaudens, France, type FPP31) after extrusion (7/8 mixing, 1/8 shear stress, Prism

Download English Version:

<https://daneshyari.com/en/article/7084249>

Download Persian Version:

<https://daneshyari.com/article/7084249>

[Daneshyari.com](https://daneshyari.com)