



# Hydrolysis and composition of recovered fibres fractionated from solid recovered fuel



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## HIGHLIGHTS

- Recovered fibres were fractionated from solid recovered fuel and characterised.
- Recovered fibres have a high content of carbohydrates and ash.
- The carbohydrates in recovered fibres can be hydrolysed to sugars with enzymes.
- The effect of solids loading and surfactants on enzymatic hydrolysis was studied.

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## ABSTRACT

Fibres fractionated from solid recovered fuel (SRF), a standardised market combustion fuel produced from sorted waste, were considered as a source of lignocellulosic fermentable sugars. The fibre yield from four samples of SRF was 25–45%, and the separated material consisted of 52–54% carbohydrates, mainly glucan, with a high content of ash (12–17%). The enzymatic digestibility of recovered fibres was studied at low and high solids loading and compared with model substrates containing only chemical and mechanical pulps. Above 80% hydrolysis yield was reached at 20% solids loading in 48 h, but variation was observed between different samples of recovered fibres. Surfactants were found to improve the hydrolysis yield of recovered fibres especially in tumbling-type of mixing at low solids loading, where hydrolysis was found to stagnate without surfactants. The results suggest that SRF is a potential source of easily digestible lignocellulosic carbohydrates for use in biorefineries.

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## 1. Introduction

Sugars are raw material for microbial and chemical processes producing biofuels such as ethanol and butanol, polymer precursors like lactic and succinic acid, and other chemicals including xylitol and furfural (Menon and Rao, 2012). In addition a vast fermentation industry producing e.g. amino acids, antibiotics and industrial enzymes is using starch- or sucrose based glucose as a

raw material. As the sustainability of using sugars from the food chain for the production of non-food products has been disputed, there is a need to find alternative, non-food sources of sugar for the non-food industry. Various lignocellulosic feedstocks could, in this respect, serve as an interesting alternative. The first commercial-scale lignocellulosic ethanol plants are currently under construction or have recently started operations (Balan et al., 2013). Corn stover, sugar cane bagasse, wheat straw and the energy crop *Arundo donax* are favoured as feedstocks in these first industrial plants. Other potential biomass sources besides agricultural residues are forest industry related biomass streams. Native woody streams like logs, harvest residues, saw dust and bark, and some more processed streams like pulp and paper mill sludges are rich in carbohydrates, but may be expensive or challenging to process. Recovered pulp and paper industry products are an alternative non-food source of sugars, which could be used for

**Abbreviations:** CBH, cellobiohydrolase; CHP, combined heat and power; EG, endoglucanase; GMO, genetically modified organism; HPAEC-PAD, high-performance anion-exchange chromatography with pulsed amperometric detection; MUL, 4-methyl-umbelliferyl- $\beta$ -D-lactoside; PEG, polyethylene glycol; SRF, solid recovered fuel; TMP, thermomechanical pulp.

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the production of transport fuels and chemicals instead of being directly combusted to energy.

Solid recovered fuel (SRF) is defined as a solid fuel prepared from non-hazardous waste meeting the classification and the specification requirements laid down in the standard EN15359 (ERFO, 2013). SRF is typically municipal solid, industrial, or commercial waste, which is homogenised and upgraded to a quality that can be traded amongst producers and users. SRF is a heterogeneous fuel with a net calorific value from 3 to above 25 MJ/kg depending on the source. The current production of SRF is circa 12 Mt/a in the EU, however, the potential is much higher, estimated at circa 70 Mt/a (Straetmans, 2010). The production of refuse-derived fuel (RDF), which is shredded and sorted but not standardised, is even larger (Rotter et al., 2011). SRF is utilised for energy production in cement kilns, coal-fired power plants, lime kilns, industrial boilers and combined heat and power (CHP) plants reducing the amount of waste going to landfill. The largest European CHP and power plant capacities for SRF are currently in Germany, Finland and Sweden (ERFO, 2013).

The lignocellulose content of SRF is a key attribute as it is the climate neutral part of the fuel (Flamme and Geiping, 2012). Astrup et al. (2009) estimated the paper content of SRF to be 54% of dry content, and reviewed the biogenic carbon content to be from 45% to 85% of the total carbon, which covers 45% of SRF dry weight. This biogenic carbon present mostly in the form of cellulosic and non-cellulosic carbohydrates offers a waste-based low-cost source of fermentable sugars alternative to agricultural residues, which are seasonal and more recalcitrant. The fractionation of carbohydrates from SRF in the form of recovered fibres, and their recalcitrance towards enzymatic hydrolysis are the key factors in producing fermentable sugars from SRF. We have recently published pilot-scale trial data on an interesting biorefinery concept consisting of the fractionation of SRF, the hydrolysis of recovered fibres, ethanol fermentation and biogas production (Kemppainen et al., 2012). What is yet to be reported are the fractionation yields of recovered fibres from SRF, their composition, and the detailed behaviour of the material in enzymatic hydrolysis compared to its pure main constituents, chemical and thermomechanical pulp. The present literature on similar concepts is typically based on experiments at low processing consistency although a relatively high processing consistency appears to be necessary for feasible conversion processes of lignocellulosics (Modenbach and Nokes, 2013). It is thus important to understand the effect of solids loading on the hydrolysis yield of a particular feedstock. Surfactants are widely used chemicals that have been shown to improve the enzymatic hydrolysis of several different types of biomass (Eriksson et al., 2002). Assessing the effect of surfactants on a new type of biomass can bring down enzyme costs in an envisioned industrial plant. This paper analyses the suitability of recovered fibres fractionated from solid recovered fuel as a source of sustainable fermentable sugars.

## 2. Methods

### 2.1. Raw materials, enzymes and chemicals

SRF samples were received from three suppliers in the United Kingdom and one supplier in Finland. A sample of pulp and paper mill fibre sludge was received from an integrated pulp and paper mill in Finland. Never-dried birch and spruce kraft pulps, received as samples from Finnish pulp mills, were mixed in ratio of 51/49 w-% for use in hydrolysis experiments. A model substrate was composed by mixing birch kraft pulp, spruce kraft pulp and Finnish spruce thermomechanical pulp in ratio of 35/34/31 w-%.

Several different enzyme mixtures were constituted from nonpurified monocomponent enzymes provided by Roal Oy, Finland. The basic enzyme mixture contained Cel7A from *Acremonium thermophilum* (cellobiohydrolase I) and Cel5A from *Thermoascus aurantiacus* (endoglucanase), and was supported by the addition of Cel6A (cellobiohydrolase II) from *A. thermophilum* or *Chaetomium thermophilum*, Cel7B (endoglucanase) from *Trichoderma reesei*, xylanase from *Nonomuraea flexuosa* or *T. aurantiacus*, mannanase from *T. reesei* and  $\beta$ -glucosidase from *T. aurantiacus* or *A. thermophilum*. Protein content of the enzymes used in Figs. 1–3 was determined by Bio-Rad protein assay (Bio-Rad Laboratories, Hercules, CA, USA), which is based on the Lowry assay using bovine gamma globulin as standard. Protein content of the enzymes used in Figs. 4 and 5 was measured using Bovine Serum Albumin as standard.

The nonionic surfactant products used in the experiments were PEG 4000 (Merck, Germany), Lutensol AT 50 Flakes (BASF, Germany), and Softanol 90 (Ineos, Switzerland). Other chemicals were analytical or technical grade depending on their purpose of use.

### 2.2. Fractionation of SRF

Fibres were fractionated from SRF in small (7.5 kg dry weight SRF treated per batch) and large pilot-scale (300 kg SRF treated per batch). Fibre batches A, B and C were produced by re-pulping the SRF samples from the United Kingdom in small pilot-scale for 20 min at 5% consistency (150 kg total mass) in a 200 L and a 11 kW batch pulper (Tampulping, Finland). The disintegrated fibre sludge was let out of the reactor through a sieve plate with 3 mm width holes. The reject was washed 3 times with a total of 130 L water. The sludges and washing waters from two pulping batches were combined, and pH was adjusted to 5 using strong sulphuric acid. The fibre sludge was dewatered in a decanter centrifuge producing a stream of recovered fibres at 34–38% dry matter content, and heat treated at 95 °C for 60 min to reduce microbial load.

Batch D fibres were fractionated from SRF from the same supplier as batch A fibres, but in large pilot-scale using a 8 m<sup>3</sup> pulping tank with a 110 kW motor. Sample E was fractionated similarly to sample D but before pH adjustment a volume of dilute pulp and paper mill fibre sludge was added to make up 25% of the dry matter of the total sludge. Heat treatment was conducted after adjustment of pH with strong phosphoric acid. The dewatering of the recovered fibres was carried out on a belt press to produce circa 320 kg (on dry) recovered fibres per batch at 43% average dry matter content.

### 2.3. Hydrolysis experiments

Hydrolysis experiments were carried out as triplicates in two experimental setups: in test tubes in a water bath under magnetic mixing, and in round plastic bottles in a rotating drum placed in a heat cabinet. Conditions for test tube hydrolysis were: 1% solids content, 3 ml total volume, 400 rpm magnetic mixing. Conditions for bottle hydrolysis were: 1–25% solids content, 50 ml total volume (40% bottle fill volume), 24 rpm of the rotating drum. The bottles were moving freely in the rotating drum. Temperature was controlled at 50 °C and sodium acetate buffer with 0.02% sodium azide was used in all experiments. Buffer strength of 50 mM was used in the test tube experiments, whereas in the bottle experiment the final buffer strength in the reaction was adjusted to 100 mM. Enzyme mixtures were dosed as mg protein per g of substrate using dosages in the range of 4–16 mg/g. Surfactants were dosed 1% w/w of the substrate dry weight and added to hydrolysis by dissolving them to the buffer. Hydrolysis was stopped by boiling

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