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*Published in:*  
Cellulose

*DOI:*  
[10.1007/s10570-024-06187-y](https://doi.org/10.1007/s10570-024-06187-y)

Published: 01/11/2024

*Document Version*  
Publisher's PDF, also known as Version of record

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*Please cite the original version:*  
Kauppi, E., Niskanen, J., Hiltunen, E., & Paltakari, J. (2024). Quantifying cellulose content in plastic-cellulose material mixtures. *Cellulose*, 31(16), 9613-9621. <https://doi.org/10.1007/s10570-024-06187-y>

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# Quantifying cellulose content in plastic-cellulose material mixtures

Emilia Kauppi · Jukka Niskanen · Eero Hiltunen · Jouni Paltakari

Received: 12 June 2024 / Accepted: 20 September 2024 / Published online: 7 October 2024  
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**Abstract** This study investigates the capabilities of various measurement techniques for quantifying the cellulose content in reject material from a carton recycling center, which consists of polyethylene, cellulose, and aluminum, along with impurities. Different measurement techniques, including Fourier Transform Infrared Spectroscopy combined with Attenuated Total Reflectance (FTIR-ATR), cellulose dissolution using cupri-ethylenediamine (CED) from plastic followed by gravimetric analysis, acid hydrolysis combined with chromatography, and Thermal Gravimetric Analysis TGA, are employed in this study. Acid hydrolysis combined with chromatography and TGA shows comparable results when compared to different techniques for analyzing pulper reject. Dissolution with CED showed also comparable results but shows higher variation than TGA or chromatography. FTIR absorbance ratio of 1025/2917 correlates with cellulose content, but it shows high variation and lacks sensitivity below 5% cellulose content in polyethylene. This limitation is

attributed to factors such as the limited measurement area (1.8 mm) and the large particle size of the cellulose and LDPE mixtures, possibly caused by inadequate grinding of LDPE. In conclusion, TGA and acid hydrolysis combined with chromatography are the most reliable for quantifying cellulose content in recycling reject, providing more consistent and accurate results than FTIR-ATR or CED dissolution methods.

**Keywords** LDPE (low-Density polyethylene) · FTIR spectroscopy (Fourier Transform Infrared Spectroscopy) · TGA (thermogravimetric analysis) · CED (Cupri-ethylenediamine) · Recycling · HPAEC (High-Performance Anion-Exchange Chromatography)

## Introduction

In response to the growing demand for increased plastic production and rising concerns about climate change, there is an urgent need to advance recycling technologies (Lahme et al. 2020; Worrel et al. 2014). While paper and paperboard boast an impressive 80% recycling rate in Europe, the recycling rate for plastic packaging lags at just 40% (An official website of the European Union, 2021). Addressing this discrepancy, the European Union (EU) introduced 2019, a pivotal rule mandating that all composite materials

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containing over 5% of plastic undergo recycling (Lahme et al. 2020).

The intricate composition of modern packaging materials is showcased by e.g. paperboard-based beverage cartons, which comprising an average of 75% paperboard, 21% plastics, and 4% aluminum (Lahme et al. 2020). The plastic component in beverage cartons, primarily low-density polyethylene, plays a crucial role in serving as a moisture barrier for packaging. Typically, this plastic is applied as thin extrusion-coated films onto the paperboard surface (Emblem et al. 2012). Additionally, the caps of beverage cartons, usually made of high-density polyethylene, contribute to the overall plastic content (Robertson 2021).

Fiber from beverage cartons is characterized as high-quality because it is made from virgin fiber and bleached chemical pulp, making it a valuable material for recycling (Emblem et al. 2021).

A typical recycling process of beverage cartons involves separating the plastic from paperboard in a hydro-pulper, where cartons are agitated with water to create a slurry by disintegrating the fiber. This slurry passes through sieves, allowing the accept (the separated fiber) to continue further into the process. The hydro-pulper reject consists of the aluminum-plastic portion of the carton (Höke and Schabel 2010). Interest in energy recovery for polyethylene-aluminum (PE-AL) waste has increased due to the high heating value of aluminum-plastic waste. However, with incineration of PE-Al waste, the PE cannot be recovered, while the aluminum portion can be recycled (Zawadiak et al. 2017). Notably, the German recycling facility Palurec specializes in handling this PE-Al reject, where low-density polyethylene, high-density polyethylene and aluminum can all be separated and further recycled (The Palurec procedure, 2021).

The fiber content in the plastic-aluminum reject presents a significant challenge for further processing. Hence, effective fiber removal is vital before the subsequent separation of aluminum and plastic.

To address this challenge and facilitate the development techniques to enhance fiber removal from plastic, it is imperative to employ suitable measurement techniques.

The heterogeneity of pulping reject material and the presence of aluminum poses challenges in measuring the cellulose content. Shadangi et al. (2023)

describe a method to determine cellulose content by heating the biomass in the presence of alkaline solution at 70–90 °C with continuous stirring and further treatment in an autoclave at 160 °C and high pressure. Cellulose content is measured after filtering the solution and weighing the residue (Shadangi et al. 2023). This weighing approach is problematic with pulping reject due to the very heterogenous material and the presence of aluminum. Therefore, an approach where cellulose selective solvent is used or a characterization method where only carbohydrates are detected are needed. The ASTM E1758-01 standard method, where carbohydrates are hydrolyzed using sulphuric acid and characterized by chromatography, is considered a potential method to analyze pulping reject (ASTM E1758-01, 2020). Haslinger et al. (2019) measured cellulose content in textile mixtures with nuclear magnetic resonance spectroscopy (NMR). The sensitivity of NMR is limited due to the low (0–10%) concentration of cellulose content, so therefore this approach is not used in this test.

In this study Fourier transform infrared spectroscopy combined with attenuated total reflectance FTIR-ATR, thermal gravimetric analysis (TGA), and cellulose dissolution using cupri-ethylenediamine (CED) followed by gravimetric determination of cellulose content and acid hydrolysis combined with chromatography, are used to measure cellulose content of 0–10% in LDPE-cellulose mixtures. CED dissolution is a technique used in pulp viscosity measurements according to ISO 5351:2010 (Pulp-determination of limiting cupri-ethylenediamine (CED) solution) (ISO 5351:2010).

This study addresses two key aspects:

## Measurement techniques

Accurate measurement techniques for determining cellulose content in LDPE and subsequent reject after hydropulping from recycling factories are examined. Various measurement techniques are compared with each other to establish their effectiveness.

## Sample preparation

The methodology for preparing samples for measurement is detailed, as it can significantly affect the measurement results.

## Experimental section

### Materials and methods

All materials used in this test are presented in Table 1.

Beverage carton sample consist of 75% paperboard and 25% polyethylene.

### Methods

Fourier Transform Infrared Spectroscopy (FT-IR) analysis with attenuated total reflectance (ATR)

For the calibration curve, eucalyptus pulp and LDPE mixtures were prepared using a Retch mill. Eucalyptus pulp and LDPE pellets were weighted to formulate composite samples with varying concentrations

of eucalyptus pulp (1.5%, 1.9%, 3.5%, 5%, 5.9%, 8.1%, and 10.2%) in LDPE. Prepared samples were ground with a Retch mill using a 0.5 mm sieve size.

The obtained powders were analyzed using a PerkinElmer FT-IR spectroscopy with an ATR diamond (Perkin Elmer), model spectrum two polymer QA/QC Analysis System.

All spectra were obtained from 32 scans with a resolution of  $4\text{ cm}^{-1}$  and absorption mode using a wave-number range of 900 to  $4000\text{ cm}^{-1}$ . Five repeated measurements per sample were performed.

### Thermal gravimetric analysis (TGA)

LDPE pellets and eucalyptus pulp were individually ground in a Retch mill using a 0.5 mm sieve size to obtain powders containing pulp at concentration of 1.6%, 3.8%, 6.9%, 10.2% and 10.3% in LDPE. Samples of 100% eucalyptus and 100% LDPE were also analyzed. Sample sizes of 10–15 mg were used in the tests. The pulp-LDPE mixtures were individually recorded by the instrument with an accuracy of  $1\text{ }\mu\text{g}$ .

TGA was performed using two different equipment: TGA Q500 V20.13 Build 39 using 60 ml/min nitrogen as the sample gas and a heating rate of  $10\text{ }^{\circ}\text{C}/\text{min}$  from 25 to  $600\text{ }^{\circ}\text{C}$ . Another instrument, NETZSCH STA 449F3, used nitrogen gas flow of 70 ml/min as the sample gas and a heating rate of  $10\text{ }^{\circ}\text{C}/\text{min}$  from 40 to  $600\text{ }^{\circ}\text{C}$ .

**Table 1** Materials and equipment used in test

Materials	Supplier
0.1 M cupriethylenediamine	VWR chemicals
Eucalyptus pulp (bleached)	UPM
LDPE pellets	Borealis Polymer Oy
Beverage carton (Tetra Rex)	Stora Enso
Reject after pulper	Stora Enso
TGA STA 449F3	NETZSCH
TGA Q500 V20.13 Build 39	TA instruments
FTIR-ATR	PerkinElmer
Phenom Pure G5 Tabletop SEM	Phenom World
Q 150 R Sputter Coater	Quorum Technologies
Precisa 205 A SCS (144)	Precisa
VWR-205AC/CAL	VWR
HPAE-PAD Dionex ICS 5000+ Thermo ISQ EC	Thermo
Systec DE 23 Autoclave	Systec
Sulphuric acid 72%	VWR
Standard Analog Shaker	VWR

### Dissolving method with cupri-ethylenediamine (CED)

A 0.1 M CED solution was used to dissolve the fiber from the plastic surface. Samples weighing 0.1–0.4 g were dried at  $105\text{ }^{\circ}\text{C}$  for 1 h according to SCAN-N 23:78. Beverage cartons and pulper reject from recycling factory A were dissolved in 50 ml of the 0.1 M CED solution, while pulper reject from recycling factory B required 100 ml due to its higher fiber content. The solutions were sealed in 50- and 100-ml plastic containers to prevent oxidation of solution and placed in a VWR Standard Analog Shaker for 25 min.

After dissolution, the solutions were filtered, and the plastic pieces were washed with water and dried for 15 min at  $105\text{ }^{\circ}\text{C}$ . The weight of the plastic pieces was measured before and after dissolution and drying.

The fiber content in the plastic pieces was calculated from the weight before and after dissolution according to Eq.1

$$\text{fiber content (\%)} = \frac{m(\text{fiber} + \text{plastic}) - m(\text{plastic})}{m(\text{fiber} + \text{plastic})} \quad (1)$$

### Scanning electron microscopy

The scanning electron microscope (SEM) imaging of the sample was conducted with a Phenom-World Phenom Pure G5 Tabletop SEM. Prior to imaging, samples were coated with Quorum Technologies Q 150 R Sputter Coater using Palladium (Pd) target and 20 s coating time. In this study, SEM was used to detect the plastic surface before and after dissolution to verify that all fibers are dissolved.

### Acid hydrolysis and chromatography

A small piece of plastic from pulper reject was measured according to ASTM E1758-01(2020) Standard Test Method for Determination of Carbohydrates in Biomass by High Performance Liquid Chromatography (ASTM E1758-01 2020). In this method, carbohydrates were hydrolyzed using 3 ml of 72% sulphuric acid for 1 h at a 30 °C water bath. The samples were stirred every 10 min. After complete hydrolysis, 84 ml of deionized water was added to the samples. Samples were filtered through a glass filter to remove the undissolved parts from the solution.

Sugar recovery standards (SRS) and sample solutions were placed in an autoclave for one hour at 121 °C. SRS contained the same sugars as the standards and were used to correct for losses due to the destruction of sugars during acid hydrolysis. After cooling the samples to room temperature, both SRS and samples were diluted to 1:25 and 1:50 in 50 ml sample bottles. A volume of 150 µl of the sample liquid and SRS was filtered through a 0.2 µm filter into an autosampler vial. Monosaccharides from the hydrolysates were analyzed using Dionex ICS-5000 high-performance anion-exchange chromatography coupled with pulsed amperometric detection (HPAEC-PAD). The column used was Dionex CarboPac PA20 and flow rate 0.38 ml/min. Water was used as the eluent, and 0.1 M NaOH was added

post-column before pulsed amperometric detection (PAD).

A calibration curve was constructed using monosaccharide solutions covering the concentration range from 1 mg/l to 50 mg/l.

All observed monosaccharides were combined to determine the total carbohydrate content in the sample (mg/L). The total amount of carbohydrate weight in the original sample needs to be calculated based on the weight in a 4% concentration solution, which had a volume of 86.73 ml. SRS samples provide correction factors for carbohydrates due to acid hydrolysis. The cellulose content in original sample piece can be calculated according to Eq.2.

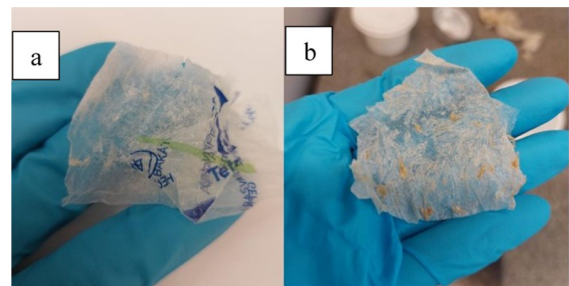
$$\text{Cellulose content \%} = \frac{\text{carbohydrates total} \times C_{\text{SRS}} \times 0,08673\text{l}}{\text{original sample weight as dry}} \quad (2)$$

Where  $C_{\text{SRS}}$  is correction factor of sugars.

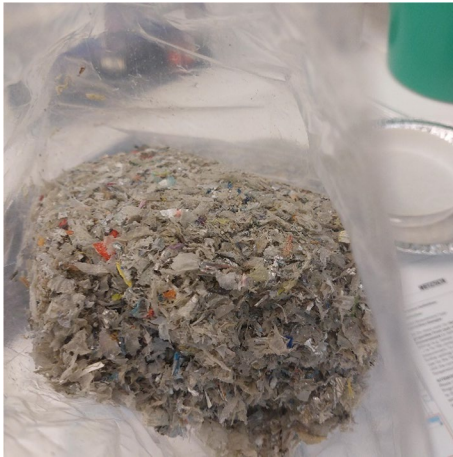
### Pulper rejects

Figure 1 shows beverage carton samples after pulping them for 1 h in laboratory pulper, alongside pulper reject from recycling factory B, which contained higher fiber content.

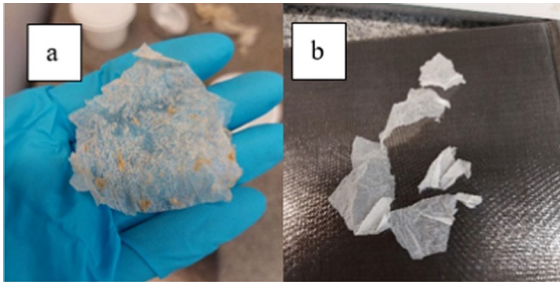
Figure 2 shows pulping reject from Recycling factory A. Reject had grayish appearance as it contained aluminum on surface.



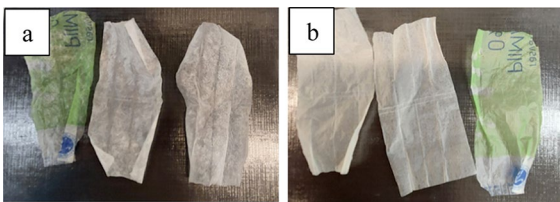
**Fig. 1** Beverage carton (Tetra Rex) sample after pulping a) and pulper reject from recycling factory B



**Fig. 2** Pulper reject from recycling factory A



**Fig. 3** Recycling reject from recycling factory B before (a) and (b) after dissolution

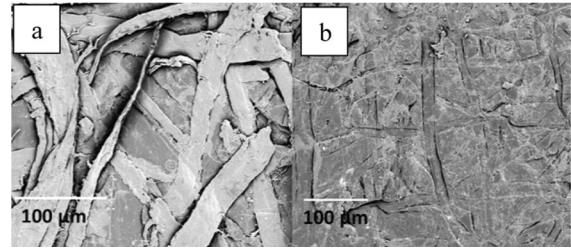


**Fig. 4** presents beverage carton sample before and after CED dissolution

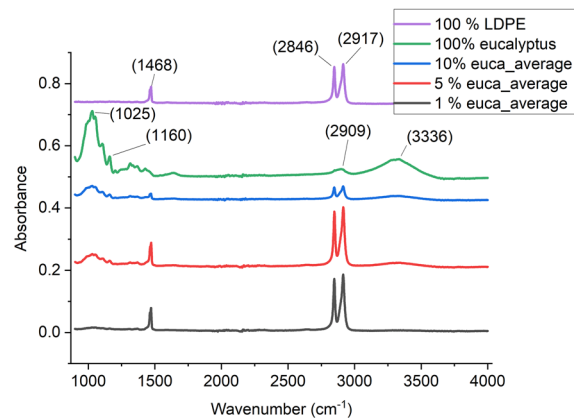
## Results and discussion

### Dissolution

Plastic pieces with fiber on their surface were collected from recycling reject, and the fibers were



**Fig. 5** Beverage carton sample after pulping and drying (a) and (b) after CED dissolution



**Fig. 6** FTIR spectra of eucalyptus powder, LDPE powder and mixtures with eucalyptus powder of 1, 5 and 10% in LDPE

dissolved in CED according to method described in Materials and Method section.

Recycling reject before and after dissolving with CED solution are presented in Fig. 3.

Visually, all fiber was dissolved from the surface after a dissolving time of 25 min.

Beverage carton samples after pulping had lower amount of fiber than recycling waste and there only 50 ml of CED was used and sample size was 0.1 g.

Figure 4 Beverage carton samples after pulping before (a) and after (b) dissolution with 0.1 M CED.

Similar to the recycling waste, all fibers were visually removed from the plastic surface after 25 min.

The samples were also inspected with SEM before and after dissolution (Fig. 5). Fibers are easily detected on the surface of the plastic. If a sample consist only a few fibers, they can be missed in visual inspection but detected in SEM.

Visual and microscopic examination confirm that the 0.1 M CED dissolution dissolves a small amount

of fiber on top of plastic surfaces. The CED solution readily absorbs onto cellulose surfaces, attributed to the interaction between cellulose and the metal complex (Zhang et al. 2019).

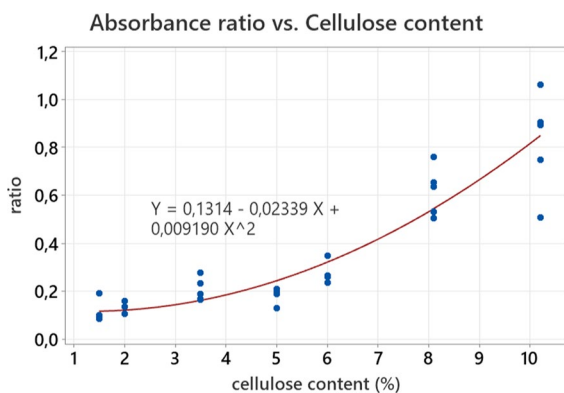
#### Fourier-Transform Infrared Microscopy (FTIR)-ATR

Mixtures containing 0–10% cellulose content in polyethylene were prepared and analyzed using FTIR-ATR. Figure 6 presents the average spectra of the samples.

Pure cellulose exhibits a characteristic cellulose fingerprint region between 900 and 1200  $\text{cm}^{-1}$ , with an absorption band at 1160  $\text{cm}^{-1}$  originating from the asymmetric C-O-C stretching between glucose units. Absorbance bands at 1025 and 1104  $\text{cm}^{-1}$  are attributed to C-O-H vibrations in the glucose units, while the band at 2909  $\text{cm}^{-1}$  is associated with cellulosic C-H. The broad band at 3300  $\text{cm}^{-1}$  is attributed to hydroxyl groups in the cellulose and bound water (Orelma et al. 2023; Yang et al. 2007).

Pure LDPE exhibits prominent bands at 2846  $\text{cm}^{-1}$  and 2917  $\text{cm}^{-1}$  corresponding to asymmetric and symmetric C-H stretching. A strong band at 1468  $\text{cm}^{-1}$  is indicative of  $\text{CH}_2$  scissoring vibration (Rajandas et al. 2012). Notably, an increase in the cellulose content is reflected in increased absorbance bands around the 900–1200  $\text{cm}^{-1}$  range, while an increase in polyethylene content is evident in 2840–2920  $\text{cm}^{-1}$  range.

The spectra presented in Fig. 6 are averages of repeated measurements. To establish a calibration curve, each sample were measured five times



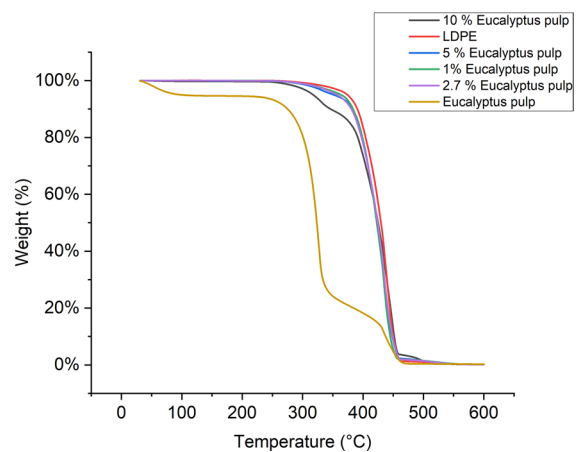
**Fig. 7** Absorbance ratio of 1025  $\text{cm}^{-1}$  to 2917  $\text{cm}^{-1}$  as a function of cellulose content

and the absorbance ratio of bands at 1025  $\text{cm}^{-1}$  and 2917  $\text{cm}^{-1}$  was determined. The data was analyzed using both absorbance band areas and the maximum of the band absorbance. Both methods yielded comparable results, hence, we chose to use the peak values of the absorbance bands to generate calibration curve. The resulting calibration curve is presented as a function of cellulose content in Fig. 7.

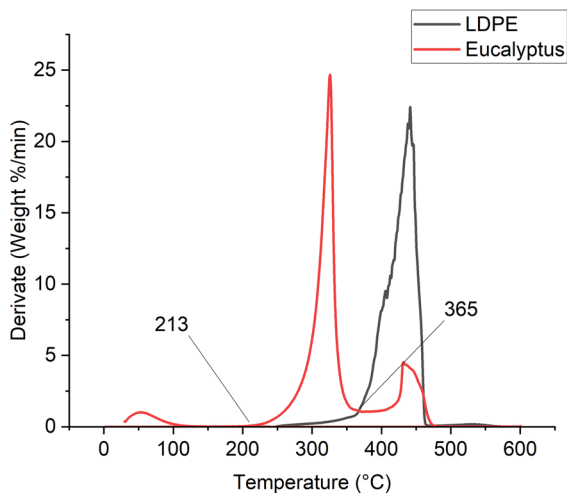
It was observed that samples with less than 5% cellulose content could not be distinguished from each other. Higher cellulose content samples have higher variation. FTIR-ATR is highly surface-sensitive, with the active area of the ATR crystal being only 1.8 mm in diameter. Given that our powder size is 0.5 mm, only a few grains of powder could fit into the measurement area.

For qualitative measurement, FTIR-ATR is fast and accurate. However, when it comes to measuring the heterogeneous cellulose content in recycling waste within such a small surface area, it proves too robust. It was considered that the LDPE and cellulose in our sample do not mix well, potentially contributing to the challenge of accurately measuring cellulose content in polyethylene within this limited surface area.

To enhance the capability of FTIR-ATR for quantitative analysis, transmittance measurements with a finer powder size is proposed.



**Fig. 8** TGA curves of 100% LDPE, 100% eucalyptus pulp, and 1–10% eucalyptus pulp contents in LDPE



**Fig. 9** Weight%/min for eucalyptus pulp and LDPE samples

### Thermogravimetric analysis (TGA)

TGA measurements were carried out for 100% eucalyptus pulp, 100% LDPE and different cellulose contents in LDPE in the range of 0–10%, as shown in Fig. 8. Weight% as a function of temperature are presented in Fig. 8.

The trace obtained from LDPE exhibited a one-step degradation profile, whereas cellulose-LDPE mixtures displayed a two-step degradation profile, indicating the presence of cellulose. The degradation

**Table 2** Cellulose content,  $t_d$ , error from actual and recorded cellulose content and residue for samples containing 0–10% cellulose in LDPE

Sample	Degradation 115–365 °C/%	$T_d$ / °C	Error	Residue
10.3% cellulose in LDPE	10.4	329	0.1	3
10.2% cellulose in LDPE	9.1	338	1.1	1.7
6.9% cellulose in LDPE	6.4	352	0.5	4.5
4.2% cellulose in LDPE	4.4	405	0.2	0.6
3.8% cellulose in LDPE	3.9	418	0.1	0.2
1.6% cellulose in LDPE	2.0	435	0.4	0.3

temperatures of the two components were easily discernible from the DTG curves (Fig. 9). Eucalyptus pulp commenced degrading at 213 °C, while the majority of LDPE exhibited degradation at 365 °C. Yang et al. (2007) concluded that hemicellulose degradation primarily occurs in 220–315 °C, whereas cellulose degradation predominantly takes place between 315 and 400 °C. Eucalyptus pulp contained both cellulose and hemicellulose.

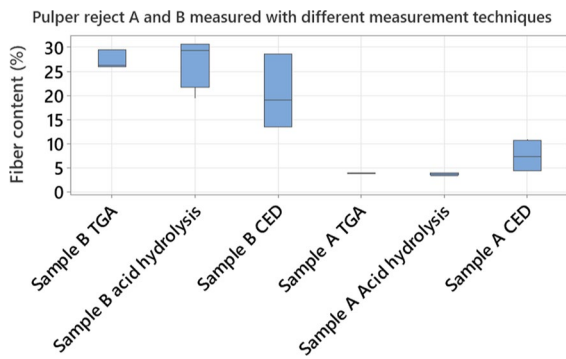
Table 2 presents cellulose content detected from TGA from mass loss at 115–365 °C. 115 °C is starting point that all moisture is left from the sample. At 365 °C, LDPE starts to decompose. In the Table 2, temperatures where a 5% weight loss occurs ( $T_d$ ), and residue weight are also presented.

We found that the cellulose content of our samples, which contained 0–10% cellulose in LDPE, can be determined from the mass loss between 115 and 365 °C TGA data with an accuracy of 98.5%. This accuracy was calculated using 6 sigma rules of normally distributed data of errors. The degradation temperature ( $T_d$ ) decreased with higher cellulose content. It was observed during measurement that even a small amount of leakage can increase the error. Additionally, it is crucial to ensure there is no contamination in the chamber or crucibles, and the chamber should be sufficiently stabilized before starting the measurement. Always perform a cleaning step after each measurement to clean the chamber and crucible. It should be noted that the char left in the crucibles after the measurements (residue) was not included in the cellulose content analysis. Error due to char could increase when measuring higher cellulose contents.

**Table 3** Chromatography average results for reject from recycling center A and B

Carbohydrate	Recycling center A mg/L	Recycling center B mg/L
Arabinose	n.a	0.89
Rhamnose	n.a	n.a
Galactose	0.46	0.66
Glucose	16.55	62.39
Xylose	1.26	9.54
Mannose	0.88	4.90
All	19.15	78.38





**Fig. 10** Boxplot charts of pulper rejects from recycling center A and B. Chart shows median values as line inside the box and box shows variation of the measurements

#### Acid hydrolysis and chromatographic measurements

Pulper rejects from recycling factories A and B were measured using acid hydrolysis combined with chromatography according to standard ASTM E1758-01(2020) as described in the methods (ASTM E1758-01, 2020).

The reject from recycling factory A was reported to have about 4–5% cellulose content. Additionally, aluminum on the surface of the plastic could be observed as grey appearances in the plastic.

Table 3 presents the average monosaccharide contents for pulper rejects from recycling center A and B as mg/L.

Each sample was measured twice. Standard samples containing 1, 10, 25 and 50 mg/L of Arabinose, Rhamnose, Galactose, Glucose, Xylose and Mannose were measured at the same time.

It was evident that reject from recycling center B contained much higher carbohydrate contents in samples. It is logical as recycling center A was designed to recycle beverage carton while the other was for Old Corrugated Containers (OCC).

Figure 10 shows the cellulose content analysis for hydropulper reject from recycling factories A and B, presented as sample A and B, respectively. For TGA analysis, sample B was sieved using a 19 mm sieve to remove some impurities from the sample and ground to a 0.5 mm powder before analysis.

Results are close to each other. Sample B had higher cellulose content than expected, and therefore the accuracy of TGA is not known. Values inside the boxplot of mean fiber content and variations were

$27.3 \pm 2.2\%$ ,  $29.8 \pm 1.0\%$ ,  $21.1 \pm 7.6\%$  measured with TGA, acid hydrolysis and chromatography, and CED, respectively. The larger variation of the CED method can be due to large variation within the sample. Additionally, it is possible that other impurities are removed during dissolution and affect the results. Sample A shows much lower cellulose content values of  $3.9 \pm 0.1\%$ ,  $3.7 \pm 0.3\%$  and  $7.5 \pm 3.5\%$ , measured with TGA, acid hydrolysis and chromatography, and CED dissolution method, respectively. Results show higher variation in Sample A using the CED method than other methods. All methods show higher cellulose content values for Sample B than Sample A. While CED can dissolve cellulose from plastic, it is not known how it affects other impurities and whether some can be washed away during dissolution. Additionally, measurements are performed using different pieces of recycling waste, and therefore variation in recycling waste can cause differences in measurements.

#### Conclusions

Acid hydrolysis combined with HPAE, TGA and the CED method yields comparable results for cellulose content when measuring reject after hydropulping. TGA and acid hydrolysis combined with chromatography provided the most similar results, while the CED dissolution method showed the most variation. Sample A included aluminum powder and other small particle impurities on the surface of the plastic, which could have led to higher results in Sample A. Impurities from the plastic surface can be removed during rinsing and CED dissolution, which would be included in cellulose content calculations.

TGA had an accuracy of 98.5% for cellulose content measurement in the range of 0–10% in LDPE. However, if the hydropulper reject contains other plastics besides LDPE, it could affect the accuracy of the results. For FTIR, a fine and homogenous mixture is needed to achieve repeatable measurements, but due to the lack of a suitable grinding tool, FTIR was not used in hydropulper reject analysis because of its lack of sensitivity below 5% cellulose content and high variation within the samples. For the CED method, the heterogenous nature of the hydropulping reject can cause variation in measurement, as

observed in this test. All tests also had different sample sizes, with TGA requiring the smallest sized sample to fit the TGA crucible.

**Acknowledgments** The authors would like to extend their gratitude to Stora Enso for providing access to their recycling factory and for the introduction to their recycling processes and laboratory measurements.

**Author contributions** Doctoral researcher Emilia Kauppi made the main contributions to this article, while the other authors provided expertise and guidance during the tests and in the writing of the article.

**Funding** Open Access funding provided by Aalto University. Additionally, we acknowledge that this work was conducted as part of the Beyond Circularity program, with Valmet serving as the leading company. Funding for this project comes from Stora Enso, Valmet and Bioinnovation center.

**Data availability** Data is in related files.

#### Declarations

**Competing interests** The authors declare no competing interests.

**Ethics approval** Not applicable. This article does not contain any studies with human participants or animals performed by any of the authors.

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