

Valorization of paper sludge for bioethanol and biogas production

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Abstract: This study evaluated the physicochemical properties and bioenergy potential of three distinct paper sludges: virgin pulp sludge (VP-PS), corrugated cardboard sludge (CR-PS), and tissue and printing paper sludge (TPR-PS). From the experimental runs, VP-PS exhibited the highest ethanol yield ($46.8 \pm 3.7 \text{ g L}^{-1}$, 87.4% conversion), which can be attributed to its high glucan content and efficient enzymatic hydrolysis. Corrugated cardboard, despite its higher lignin content, demonstrated superior biogas and methane production ($94.4 \pm 8.3 \text{ L kg}^{-1}$ and $54.4 \pm 5.1 \text{ L CH}_4 \text{ kg}^{-1} \text{ TS}_{\text{fed}}$), likely due to its elevated xylan levels and favorable bulk density. The TPR-PS was characterized by high ash content and showed lower performance for both bioenergy production pathways but displayed improved solid handling due to its higher bulk density and lower water-holding capacity. These results provide a good explanation of the potential bioconversion pathways for the different paper sludge characteristics to maximize bioenergy yield.

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Key words: paper sludge; anaerobic digestion; fermentation; bioenergy; bioethanol; bulk density

Introduction

Paper production is projected to reach 550 million tonnes per year globally by 2050, a 38% growth from current production levels.¹ Leading pulp and

paper producing countries like South Africa have shown significant growth in the pulp and paper mills and a corresponding increase in paper sludge volumes, estimated at 500 million tonnes of wet paper sludge (PS) annually.² Depending on the source and composition of the raw

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material used, paper sludge can be categorized as corrugated recycled sludge (CR), tissue and printing paper recycled sludge (TPR), or virgin pulp (VP) sludge.¹ Paper sludge typically has a high moisture content (50% to 80%) and is rich in fibers and ash, with small amounts of heavy metals.^{3,4}

Current paper sludge disposal practices involve dewatering followed by either landfilling or incineration.⁵ These unsustainable approaches are associated with substantial costs and have negative environmental impacts.⁶ The need for cost-effective and sustainable alternatives is therefore evident. To address this challenge, the South African government has implemented legislation that restricts the landfilling of this waste, intensifying the need for alternative waste management strategies within the industry.⁷ Recent legislation has introduced prohibitively high gate fees, imposed carbon tax on organic waste, and forbidden the landfilling of waste with moisture content exceeding 40%.⁸ The need for innovative approaches for resource recovery and/or reuse is therefore necessary for the sustainable development of the pulp and paper industry.

The production of biofuels (biomethane, biohydrogen, and bioethanol) from lignocellulosic biomass has attracted significant global interest due to its potential as a sustainable alternative to fossil fuels. Valorization of paper sludge provides the benefits of sustainable waste management and also economic value from the new product, allowing for sustainability in the pulp and paper industry.⁹ Despite technological advances, the commercial-scale production of biofuels remains economically challenging, primarily due to the complex and recalcitrant structure of lignocellulosic feedstocks, which hinders efficient hydrolysis.^{10,11}

Paper sludge offers several advantages including a minimal need for pretreatment before enzymatic hydrolysis, zero or negative biomass cost, and the possibility of direct integration into existing industrial infrastructures such as paper mills.¹² These advantages allow for production within the price point, increasing market competitiveness of biofuels derived from paper sludge in markets already saturated with conventional products.¹³ These attributes position this waste stream as a promising feedstock for second-generation biofuel production and an attractive option for promoting circular bioeconomy initiatives in the paper and pulp industry.

This study therefore investigated the potential of paper sludge valorization for bioethanol and biomethane production by simultaneous saccharification and fermentation (SSF) and anaerobic digestion, respectively, on a pilot scale. The study used optimized enzyme-dosing and solid-loading process parameters provided by earlier optimization studies by Boshoff *et al.*² and Robus *et al.*¹⁴ for

bioethanol and biomethane production, respectively, from paper sludge.

Material and methods

Feedstock preparation and characterization

Feedstock preparation

Samples of the paper sludge used were collected from three major representative paper and pulp mill operation companies in South Africa. These included corrugated recycle paper mills (CR-PS) from Mpact Felixton, tissue and printing recycling mills (TPR-PS) from Twincare Bellville, and virgin pulp mills (VP-PS) from Mondi Richards Bay. A fresh and representative (homogeneous) sample from the production lines was collected and transported in plastic drums. The three paper sludge samples were initially dried in a greenhouse to a moisture content of approximately 15%. Once dried, the material was homogenized through subsampling using the cone and quarter method. The homogenized samples were then ground using a Drottsky S1 hammer mill, equipped with a 2 mm screen, to achieve a consistent particle size. A portion of the milled paper sludge was pelletized using an MPEL200 pelletizer from ABC Hansen Africa; to produce dense pellets with a diameter of 6 mm. The prepared paper sludge samples were all kept in sealed plastic bags at room temperature until further use.

Feedstock characterization

Fresh paper sludge samples were characterized for glucan, xylan, lignin, extractives, and ash following the standard biomass characterization protocols developed by the National Renewable Energy Laboratory (NREL).¹⁵ The fresh samples of paper sludge were also analyzed for bulk density and water-holding capacity. The bulk density measurement was carried out for dry, milled, and pelletized paper sludge as well as residues from fermentation and anaerobic digestion. The difference between the dry fresh paper sludge and the milled/pelletized assessment measured the impact of the mechanical pretreatment whereas the difference between the dry paper sludge and the residues represented the impact due to biochemical treatment.

Bulk density was measured by filling a 100 mL beaker with oven-dried paper sludge (dried at 105 °C for at least 24 h) to the 100 mL mark. The filled beaker was weighed and the bulk density was calculated as the ratio of the dry paper sludge weight (kg) to the volume of the beaker (m³). The water-holding capacity (WHC) was determined following a method

described by Boshoff *et al.*² with modifications. Briefly, 1 g of the oven-dried paper sludge sample was mixed with 10 mL of water at 20 °C in weighted conical tubes and allowed to soak to saturation completely for at least 24 h. The conical tubes were centrifuged in a centrifuge with rotary spinner at 2500 rpm and the supernatant decanted. The water-holding capacity was calculated as the ratio of the weight of water absorbed to the dry weight of the dry sample of the paper sludge.

Experimental setup

Fermentation experiments

Enzyme, yeast, and substrate preparation

The enzyme cocktail used for hydrolysis comprised Viscamyl Flow (Danisco Genencor, Brugge, Belgium) and Novozym 188 (Novozymes, Bagsvaerd, Denmark) mixed in a 10:1 (v/v) ratio. The activity of Viscamyl Flow was 140 FPU mL⁻¹ and that of Novozym 188 was 929 IU mL⁻¹, determined using the method described by Dashtban *et al.*¹⁶

An industrial yeast strain, *Saccharomyces cerevisiae* MH100, was used for fermentation. The strain was obtained from fresh glycerol stocks stored at -85 °C in the culture collection in the Department of Microbiology at Stellenbosch University. Yeast was propagated in a growth medium containing 20 g L⁻¹ glucose, 20 g L⁻¹ peptone, and 10 g L⁻¹ yeast extract for at least 18 h prior to use – sufficient to reach the late exponential growth phase.

Each paper sludge sample for fermentation was autoclaved separately at 121 °C for 15 min at a 10% dry solids loading (based on working volume) and allowed to cool for at least 24 h before use.

Fermentation setup

Simultaneous saccharification and fermentation (SSF) experiments were conducted in a fed-batch model using 20 L baffled bioreactors (New Brunswick Scientific, Edison, NJ, USA), with a working volume of 10 L. Figure 1 summarizes the experimental setup and process flow. The reactors were fitted with three Rushton impellers for efficient mixing. *In situ* sterilization of the reactors and fermentation medium containing 3 g L⁻¹ corn steep liquor and 0.62 g L⁻¹ magnesium sulfate heptahydrate, was conducted at 121 °C while agitated at 150 rpm. The total volume of the fermentation medium was calculated as the working volume (10 L) minus the combined volume of enzyme, yeast, and total solids. Reactors were allowed to cool to 37 °C prior to the start of fermentation.

With the fermentation media in the reactor, 3% (w/v) dry solids of paper sludge were added, along with the 5% (w/v) of yeast inoculum. The enzyme cocktail was applied at 15 FPU g⁻¹ dry substrate for tissue and printed recycled paper sludge (TPR-PS), 11 FPU g⁻¹ dry substrate for corrugated recycled paper sludge (CR-PS), and 20 FPU g⁻¹ dry substrate for virgin pulp paper sludge (VP-PS). These dosages were based on the optimization results of Boshoff *et al.*² and Robus

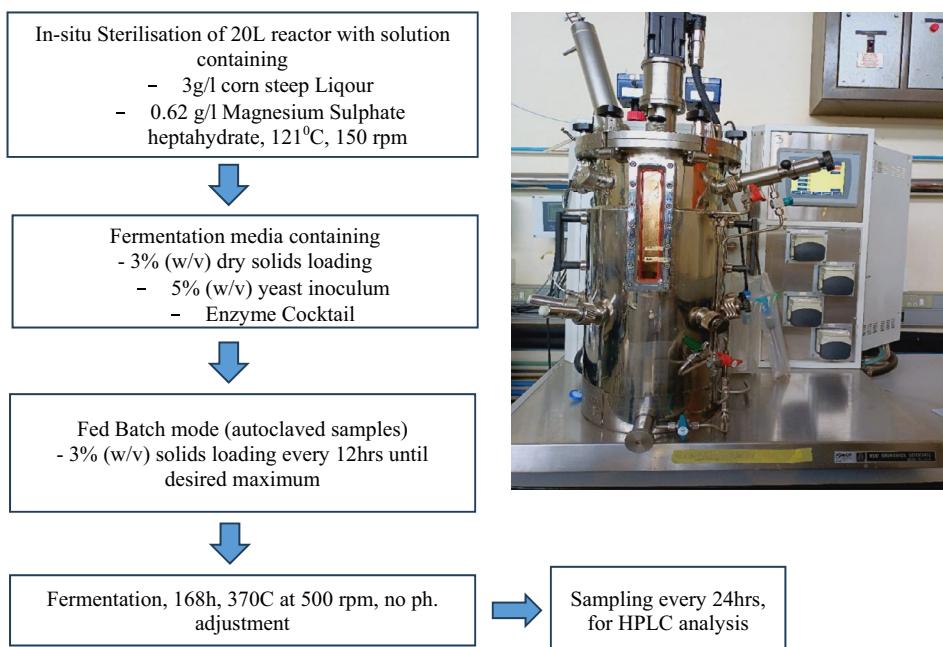


Figure 1. Fed-batch simultaneous saccharification and fermentation (SSF) experimental process flow setup and the 20 L benchtop reactor used for ethanol production.

*et al.*¹⁴ Boshoff *et al.*² reported optimum enzyme dosages of 11 FPU g⁻¹ dry solids at 27% (w/w) solids loading for CR-PS and 20 FPU g⁻¹ dry solids at 18% (w/w) solids loading for VP-PS. Robus *et al.*¹⁴ reported an optimum enzyme dosage of 15 FPU g⁻¹ dry solids at 21% (w/w) solids loading for TPR-PS. Paper sludge was fed into the reactor in fed-batch mode at 3% (w/v) every 12 h until the target solids loading was reached. The total solids loading was 330 g L⁻¹ for TPR-PS, 270 g L⁻¹ for CR-PS, and 180 g L⁻¹ for VP-PS.

The fermentation was conducted for 168 h at 37°C with agitation at 500 rpm and without pH adjustment. Samples were collected every 24 h for analysis of sugar and ethanol concentrations. These concentrations were determined using Dionex UltiMate 3000 (California, USA) high-performance liquid chromatography (HPLC) equipment with a Biorad HPX-87 H column and a refractive index detector. Samples were first centrifuged equipped with a rotary spinner at 10 000 rpm for 10 min to remove suspended solids, and the supernatant was filtered through a 0.22 µm syringe filter. Where necessary, samples were diluted with deionized water to ensure that ethanol concentrations fell within the calibration range. Ethanol was identified and quantified by comparing the retention time and peak areas with those of known ethanol standards ranging from 0.1 to 10 g L⁻¹. Quantification was performed using a standard calibration curve, and quality control was ensured by regular analysis of blanks and standards, as described by Avila *et al.*¹⁷

Anaerobic digestion experiments

Inoculum and substrate preparation

The inoculum was sourced from a wastewater treatment plant of a brewery facility in South Africa. It was initially allowed to settle to concentrate the solids, after which the supernatant was carefully decanted into a clean vessel. To activate the microbial community and eliminate any remaining substrates, the inoculum was incubated under anaerobic conditions at 37°C with gentle agitation at 93 rpm for a period of 2 weeks.

Anaerobic digestion set-up

Batch anaerobic digestion experiments were conducted using custom-designed 30-L continuously stirred tank reactors (CSTRs) operated at a constant temperature of 37°C with intermittent mixing at 93 rpm. Each digester was equipped with a lid housing several components, including a motor, feed inlet funnel, temperature sensor, level gauge, and a gas outlet valve connected to a gas flow measurement system. Mixing was achieved using a central shaft fitted with a Rushton-type impeller driven by the motor, as shown in Fig. 2. The reactors were double jacketed, with the outermost

jacket fitted with insulation material for thermal regulation. The inner jacket, containing circulating water, was used for temperature control and was equipped with an inlet port for replenishing water levels when necessary. Liquid sampling and drainage ports were positioned at the base of the vessels. Sensor data for temperature and gas output were collected using a data acquisition interface.

The experiments were conducted at a working volume of 21 L. This represented 70% of the digester's working volume. The total solids loading was 10% (w/v) for both TPR-PS and CR-PS substrates, and VP-PS was prepared at 6% (w/v). This was because of the differences in bulky densities for TPR-PS and CR-PS compared to VP-PS (Fig. 3(a)). The difference in total solids loading balanced out the total mass in the digester for any paper sludge type to within 10% of the total working volume, as required for the setup. A 10% (w/w) inoculum was added to each of the reactors. Experiments were run in duplicate for 30 days. The volume of biogas generated was determined by the displacement of water in the manometer, providing a direct measurement of gas production, as summarized in Fig. 2. Gas samples were collected in Tedlar bags from each digester every 7 days and analyzed. The collected gas was analyzed in duplicate for methane and carbon dioxide using a Compact GC^{4.0} gas chromatograph (GC) from CE Elantech, NJ, USA. helium and argon were used as carrier gases at a flow rate of 5.0 mL min⁻¹ and a reference gas flow rate of 1.0 mL min⁻¹.

Results and discussion

Paper sludge characterization

Table 1 presents the results of the composition analysis. The VP-PS demonstrated the highest glucan concentration at

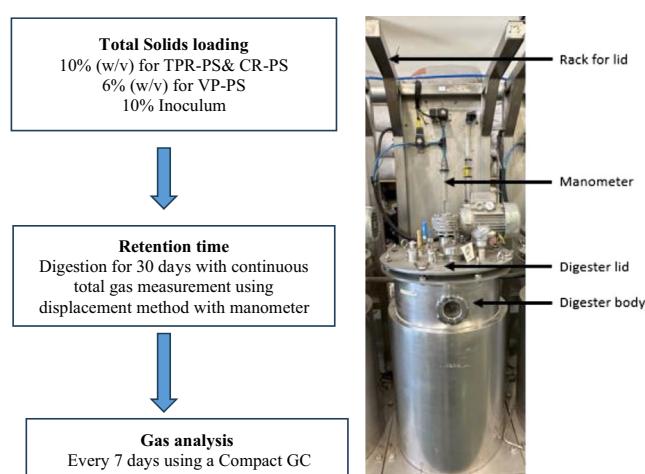


Figure 2. Anaerobic digestion experimental process flow and the 30 L digester used for the pilot scale runs.

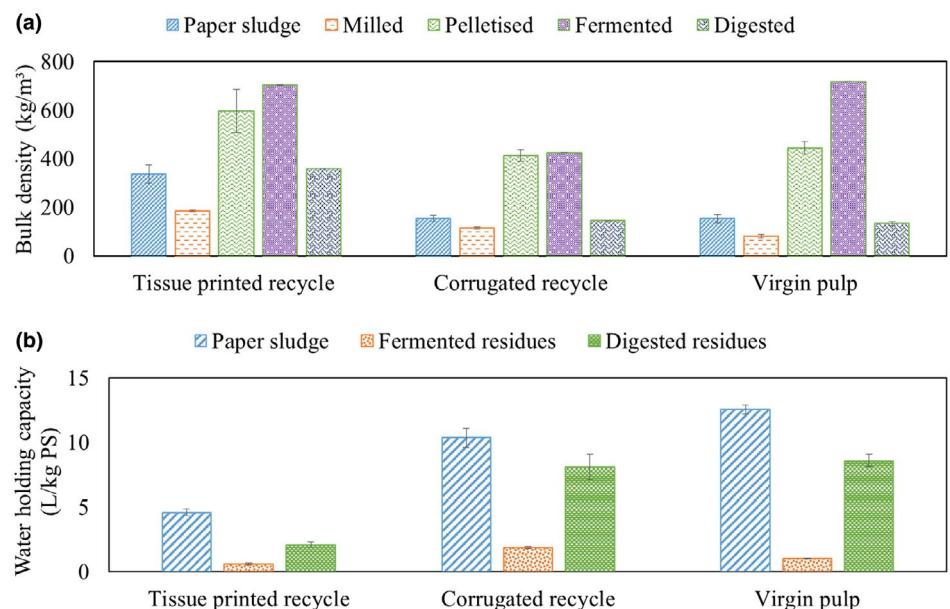


Figure 3. (a) Bulk density and (b) water-holding capacity of paper sludge after mechanical and biochemical treatment.

Table 1. Composition of paper sludge.

Parameter (% w/w)	Paper sludge		
	Tissue and paper	Corrugated carboard	Virgin pulp
Glucan	20.8±0.1	37.5±0.4	52.0±0.4
Xylan	4.9±0.2	13.0±1.1	10.6±0.4
Lignin	6.4±0.1	13.1±0.1	5.1±0.1
Extractives	5.1±0.1	10.4±0.1	7.4±0.1
Ash	62.9±0.4	25.9±0.3	24.8±0.1

52.8% (w/w), and this can be attributed to chemical residues from chemicals used for decontamination in Kraft mills.² The CR-PS had the highest xylan (13% w/w), lignin (13.1% w/w), and extractive (10.4% w/w) fractions (Table 1). High xylan content makes the CR-PS a better substrate for anaerobic digestion (AD) and fermentation followed by VP-PS (10.6% w/w) and TPR-PS (4.9% w/w). Paper sludge from corrugated paper exhibited on average more than twice the lignin content of the other two paper sludges (Table 1), reflecting the use of mechanical pulping in corrugated paper recycling mills, which retains most of the lignin in the pulp. In contrast, Kraft pulping removes nearly all lignin during processing.¹⁸

The ash content of the three paper sludges was between 24.8% and 62.9% (w/w) (Table 1). The lowest amount of ash was observed in the VP-PS sample. This was expected because the sludge came from virgin pulp with minimal foreign material. The TPR-PS presented the highest ash content. This can be attributed to the fact that the plant used recycled material that comprised mainly calcium carbonate from the ink and fillers.¹⁸ In this study, the CR-PS sample

exhibited a higher ash content than the levels documented by Boshoff *et al.*² for similar paper sludge material. This difference suggests that the chemical composition and characteristics of paper sludge waste can differ between mills, depending on the specific pulping method used at the time of production.

The TPR-PS exhibited the highest density at 340 kg m⁻³, whereas both CR-PS and VP-PS recorded lower values of 160 kg m⁻³ (Fig. 3(a)). The higher bulk density of TPR-PS may be attributed to its greater filler and ink residue content, which contributed to a denser composition despite the fibrous nature of the material. Pelletizing of the paper sludge increased the bulk density by 65% for VP-PS. A reduction in density saves reactor space for biochemical processing operations, allowing for better mixing.^{19,20} Residues from the fermentation process showed the highest bulk density. This can be attributed to the remaining lignin and ash left behind after fermentation, with very small amounts of fibrous material. This reduction in bulk potentially reduces the handling costs involved in the sludge disposal due to reduced

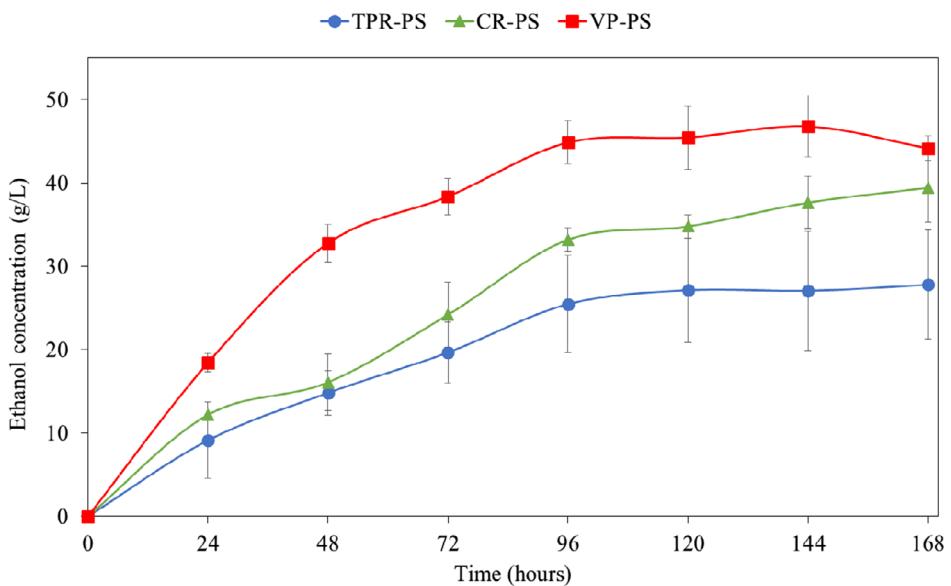


Figure 4. Ethanol yield from paper sludge fermentation for tissue and printed paper recycled sludge, corrugated cardboard recycled sludge, and virgin pulp sludge.

volumes. The density of the digested residues was similar to the density of undigested paper sludge. This was due to the presence of the undegraded fiber that remained after the digestion process.²¹

Figure 3 shows that VP-PS had the highest water-holding capacity. The enhanced water retention in VP-PS can be linked to the longer fibers typically produced through chemical pulping processes in virgin pulp processing.² It is difficult to dispose of a sludge with a high water-holding capacity, even in landfills, because of the need for dewatering prior to land filling, which is an energy intensive – and thus costly – process.²² The lower water-holding capacity observed in TPR-PS can be attributed to its high ash content and reduced fiber levels. The fillers and ink commonly found in the TPR-PS are hydrophobic in nature, which likely hinders the material's ability to retain moisture.²³

Residues from the fermentation and anaerobic digestion processes for VP-PS reported a water-holding capacity of 1.0 and 8.2 L kg⁻¹ respectively, representing 92% and 32% respective reductions from the fresh sludge. The difference in reported percentage reduction can be attributed to the retained fiber structure, which remains undegraded after anaerobic digestion but fully utilized or destroyed after fermentation.²¹ In a similar study, Boshof *et al.*² also recorded notable reductions in WHC after fermentation, reporting decreases of approximately 60% for CR-PS and 47% for VP-PS. These findings support the observations that fermentation can lead to a more extensive structural breakdown of the paper sludge.

Paper sludge ethanol and methane yield

Ethanol yield

Figure 4 shows the results for the ethanol yields. Among the three paper sludges studied, VP-PS yielded 46.8 g L⁻¹ of ethanol, the highest among the three sludges, and this corresponded to 87.4% of the theoretical yield. The sludge also presented the highest process productivity at 0.325 g L⁻¹ h⁻¹. This performance can be attributed to the high enzyme dosage (20 FPU g⁻¹ PS) combined with the low solids loading (180 g L⁻¹), which facilitated efficient enzyme diffusion and hydrolysis. Although the hydrolysis rate declined after 48 h due to cellulose depletion, VP-PS remained the most effective substrate. Similar trends have been reported previously, with low solid loading and high enzyme concentrations improving ethanol yield from Kraft paper sludge.

In a similar study, batch fermentation of Kraft paper sludge with a solids loading of 135 g L⁻¹ and an enzyme dosage of 22 FPU g⁻¹ sludge achieved a conversion efficiency of 75% and ethanol concentrations of up to 26 g L⁻¹.²⁴ The results achieved by Kang *et al.*²⁴ are similar to those reported in the present study, although this study showed a higher conversion efficiency. This can be attributed to the fed-batch model used in this study and the corresponding lower solids loading, which improved the efficiency of the enzymatic hydrolysis process significantly.^{17,25}

The CR-PS sludge achieved an average ethanol concentration of 39.4 g L⁻¹, corresponding to a conversion efficiency of 65.7% and an hourly productivity of 0.235 g L⁻¹

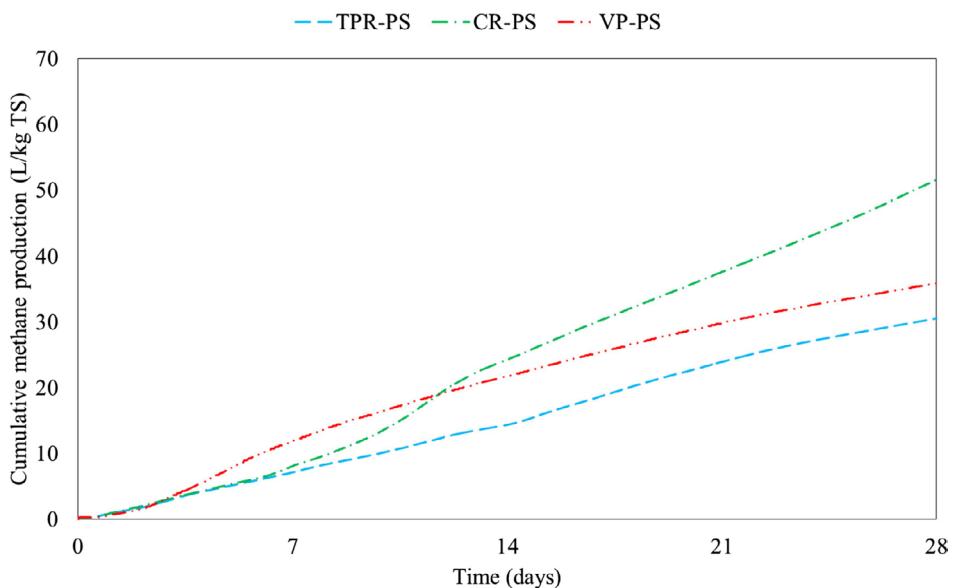


Figure 5. Cumulative methane production during anaerobic digestion for the three paper Sludges.

(Fig. 4). Although performance exceeded that of TPR-PS, the high lignin content and relatively low enzyme dosage (11 FPU g⁻¹ PS) constrained enzymatic hydrolysis efficiency. The low bulk density (413 kg m⁻³) and high water-holding capacity (10.4 L kg⁻¹) resulted in a highly viscous slurry, which impaired mixing and reduced the availability of free water required for effective enzyme activity.²⁶ These factors likely contributed to reduced sugar conversion. The results indicate that higher enzyme dosages could improve hydrolysis by minimizing the impact of the process inhibitors arising from the viscosity and lignin in the substrate. However, this would increase production costs substantially.²⁷

The TPR-PS, despite tolerating a high solids loading of 330 g L⁻¹ owing to its high bulk density (590 kg m⁻³) and low water-holding capacity (4.6 L kg⁻¹) (Fig. 3(b)), produced the lowest average ethanol concentration of 27.8 g L⁻¹. This corresponded to a conversion efficiency of 70.6% and a productivity of 0.165 g L⁻¹ h⁻¹. The low ethanol yield is attributed primarily to the high ash content (63% w/w), which has been reported to bind irreversibly to cellulase, reducing enzyme effectiveness and glucose availability.²⁴ Overall, although TPR-PS offered high solid tolerance, its low sugar content and enzyme inhibition made it the least effective for ethanol production among the three types of paper sludge.

Biogas and methane yield

Figure 5 presents the cumulative biogas and methane yields for the three types of paper sludge. Average total biogas production was 62.9 L kg⁻¹ for tissue and printed recycled paper sludge (TPR-PS), 94.4 L kg⁻¹ for corrugated recycled

paper sludge (CR-PS), and 83.9 L kg⁻¹ for virgin pulp paper sludge (VP-PS). Statistical analysis indicated no significant differences among the substrates ($P=0.063$), although CR-PS produced the highest cumulative biogas and methane yields.

The average methane yields based on the total solids fed were calculated as 31.6 L CH₄ kg⁻¹ TS for TPR-PS, 37.2 L CH₄ kg⁻¹ TS for VP-PS, and 54.4 L CH₄ kg⁻¹ TS for CR-PS. Statistical analysis showed that the average methane yield was significantly different among the three paper sludges ($P=0.026$). Notably, TPR-PS and VP-PS delivered relatively close results, which was unexpected given the high ash content (63% w/w) typically associated with TPR-PS. This observation may be explained by the high bulk density and reduced water-holding capacity of TPR-PS, which might have enhanced substrate accessibility and enzymatic breakdown during the hydrolysis phase, as noted by Bensmann *et al.*²⁸

Final methane yields normalized to volatile solids were 85.0 L CH₄ kg⁻¹ VS for TPR-PS, 73.5 L CH₄ kg⁻¹ VS for CR-PS, and 49.4 L CH₄ kg⁻¹ VS for VP-PS. In contrast, Bayr and Rintala²⁷ reported substantially higher production of 210 L CH₄ kg⁻¹ VS, from Kraft mill sludge. The discrepancy may be attributed to the longer hydraulic retention time of 45 days in their study, in comparison with the 28 day period applied in the present anaerobic digestion study.

Conclusion

This study revealed substantial differences in the biochemical and physical properties of paper sludge from corrugated cardboard, virgin pulp, and tissue and printing paper. These

differences influenced suitability for biochemical conversion processes directly, including ethanol fermentation and anaerobic digestion. Low bulk density and high water retention hindered enzymatic hydrolysis during fermentation, whereas ash content did not adversely affect anaerobic digestion. Corrugated cardboard paper sludge produced the highest methane yield, whereas virgin pulp paper sludge was the most effective substrate for bioethanol production. Overall, the results demonstrate the potential of Kraft paper mill sludge as a feedstock for bioenergy production, if bioconversion strategies are matched to specific sludge characteristics.

South Africa hosts several large pulp-and-paper mills in KwaZulu-Natal, Mpumalanga, and the Western Cape provinces. These mills generate significant quantities of paper sludge. Adapting these bioconversion processes to South Africa's pulp-and-paper industry presents a practical opportunity to enhance waste valorization and contribute to the country's renewable energy targets. Based on this study's findings, the choice of the process to deploy to an industry should be guided by sludge characteristics. The results are also applicable to other regions with similar pulp and paper industries.

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Conflict of interest

The authors declare no conflict of interest.

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