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Anaerobic Biodegradability Potential of RCF-Based Kraft Paper Mill Effluent

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ABSTRACT

The present study collected anaerobic sludge from a paper mill operating a UASB reactor on agro-based raw material (wheat straw) washings for biogas production. After determining the sludge profile and methanogenic activity of anaerobic sludge, it was further used to determine the anaerobic biodegradability of RCF-based kraft paper mill effluent. The sludge profile of collected anaerobic sludge was found w.r.t. Suspended Solids (SS) 60 $g.L^{-1}$, Volatile Suspended Solids (VSS) 23 $g.L^{-1}$, Inorganic content 62% & Organic content 38%. The presence of effective microbes in anaerobic sludge was confirmed by SEM (Scanning Electron Microscope). Degradation of organic matter present in effluent by anaerobic digestion leads to the production of biogas (methane & CO_2), a renewable energy source. The sludge profile and methanogenic activity findings supported the anaerobic treatment of kraft paper mill effluent and were positive. The methanogenic activity of anaerobic sludge was determined as 0.832 $gCOD.gVSS^{-1}$×Day, and the average anaerobic biodegradability of RCF-based kraft paper mill effluent was found to be 71.5%.

INTRODUCTION

RCF-based paper mills use waste paper as fibrous raw material for manufacturing paper and paper boards. In India, Recycled Fiber (RCF) based paper mills use indigenous (Fig. 1) and imported waste paper (Fig. 2) in different ratios as per product quality on market demand. RCF-based kraft paper-making process includes waste paper slushing, pulp cleaning & screening, pulp refining, stock preparation, and sheet formation.

Most water requirement is met by reusing and recycling backwaters in a closed loop. Fresh water is used only in sections like paper machine showers, steam generation, chemical preparation, etc. The schematic flow diagram of the process (Fig. 3).

Most wastewater generated by paper industries is rich in various biopolymers like starch, cellulose, hemicelluloses, glucose, lignocelluloses, etc. These biopolymers can be converted into sustainable biofuels, i.e., ethanol, butanol, biodiesel, biogas, hydrogen, methane, biohythane, etc., through its bioremediation by anaerobic digestion (Bhatia et al. 2020). To accomplish both of these obligations, the utilization of wastewater should be done in such a manner so that the process used would treat the wastewater along with the production of some cherished products which can be reutilized further (Reungsang et al. 2016, Ijaz et al. 2016). Using effluent/ wastewater for energy generation is economical, as this does not require expensive processes. While several emerging technologies contribute to the wastewater resource recovery challenge, biological approaches give the greatest promise to recover essential resources from effluent efficiently (Bhatia et al. 2020). Moreover, fossil sources are very limited and may deplete in the future, so alternative energy sources must be developed. Therefore, the best approaches include using wastewater to produce energy products like bioethanol, biogas, biodiesel, etc., which can be transformed into electricity (Kassongo & Togo 2011).

BMP, anaerobic biodegradability, and digestibility are similar. They all deal with the degradation of organic matter by microorganisms in anaerobic conditions. At high gas production, less organic matter remains after digestion, indicating higher biodegradability (Lesteur et al. 2010). The BMP value can be used as an index of the anaerobic biodegradation potential. The BMP is the experimental value of the maximum quantity of methane produced per gram of volatile solids (VS). The BMP is measured with the BMP test, which consists of a respirometric test, i.e., measuring the methane or biogas produced by a known quantity of waste in a batch in anaerobic conditions (Hansen et al. 2003).

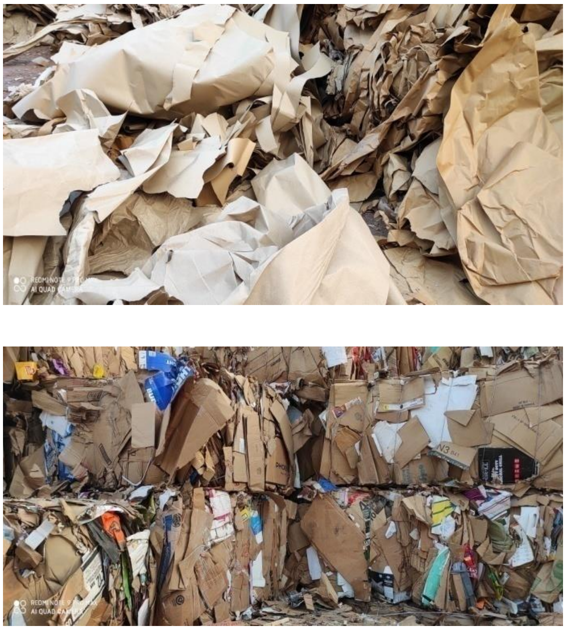

Fig. 1: Indigenous fibrous raw material.

Fig. 2: Imported fibrous raw material.

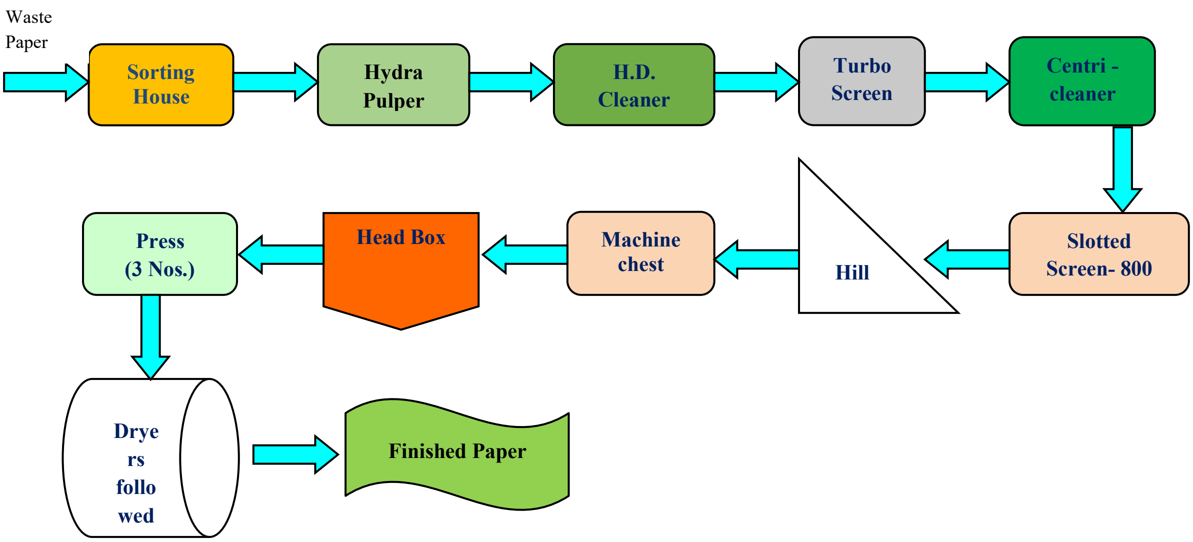

Fig. 3: Layout of the manufacturing process for kraft paper.

Overall Objective of the Study

Anaerobic biodegradability potential of effluent generated in RCF-based kraft paper mills.

Specific Objectives of the Study

(1) Characterization of effluent collected from RCF-based kraft paper mills.

(2) Determination of sludge profile of anaerobic sludge.

(3) Determination of the methanogenic activity of anaerobic sludge.

MATERIALS AND METHODS

The RCF-based kraft paper mills visited to manufacture unbleached kraft paper from waste paper (indigenous &

imported) and chemical additives like starch as sizing chemical, Poly Aluminium Chloride (PAC) as retention aid, etc. The paper-making process is water intensive; water requirement depends on pulp and stock consistency to be maintained in different unit operations. As the mills manufacture unbleached kraft paper and board, almost all unit operations are usually completed with backwater circulating in a closed loop. Grab effluent samples were collected from the mills for physical-chemical analysis per BIS/APHA standards.

Anaerobic Sludge Profile

Sludge profile is the determination of suspended solids, volatile suspended solids, and organic & inorganic constituents in the anaerobic sludge. As given in BIS and APHA (2017) standards, the gravimetric method determined the suspended and volatile solids. The anaerobic sludge collected was first washed with tap water and siphoned the supernatant water after settling the sludge (Fig. 4 & Fig. 5).

Methanogenic Activity of Anaerobic Sludge and Biodegradability of the Effluent

A sludge activity test is usually carried out in batch experiments where a fixed amount of substrate serves as feed for a predetermined amount of sludge. The specific sludge activity is estimated from the methane production rate or the substrate depletion rate and the amount of sludge present. It is usually expressed in g COD.gVSS^{-1}×day. Therefore,

$$\text{Sludge activity } (\text{g COD.gVSS}^{-1} \times \text{day}) =$$

$$\frac{\text{maximum methane production rate } (\text{L/day})}{\text{Amount of sludge } (\text{in g VSS})} \times 2.6$$

$$= \frac{y/x \times 24 \times 2.6}{\text{Amount of sludge } (\text{in g VSS})}$$

Where,

y/x is the maximum gas production rate where the curve is steepest.

2.6 is the factor, i.e., 1 L of methane produced corresponds with 2.6 g of COD removed

The assay is performed to determine the specific methanogenic activity of anaerobic sludge and the biodegradability of the effluent. It measures the potential in anaerobic sludge to convert the organic matter into biogas (methane). Where biodegradability is the measure of total methane production and COD removal efficiency from certain wastewater. In the biodegradability test, COD reduction is determined via gas production, so one or more blanks should always be incubated. For blank, sludge with all chemicals added, but with tap water instead of wastewater. The gas produced by the blank (from COD present in the sludge and deterioration of the sludge) should be subtracted from the samples with an equal amount of sludge. The anaerobic biodegradability of wastewater is calculated as follows:

$$\text{Biodegradability } (\%) = \frac{(\text{Gas}_X - \text{Gas}_B)}{\text{COD}_X} \times 260$$

Where,

Gas_X is the amount of CH_4 produced by wastewater at the end of the test, L

Gas_B is the amount of CH_4 produced by blank at the end of the test, L

Fig. 4: Collected anaerobic sludge.

Fig. 5: Washed sludge dewatering by siphoning.

COD_X is the amount of total COD added to perform the experiment.

260, is the conversion factor CH_4 produced by COD

The methanogenic activity of sludge and biodegradability is dependent on many factors. The real activity potential of sludge can be measured when ideal conditions are provided. The factors affecting are as under:

Temperature

The temperature plays an important role in the activity of sludge. Normally 30°C is recommended to measure the activity of sludge. The sludge activity can be measured at the same temperature we expect in practice.

The Concentration of Substrate and Sludge

The concentration of substrate in the micro-environment of the methanogenic bacteria is critical. The ranges of concentration of substrate & sludge are given below (Table 1).

Activity Period

The activity period is the time period when the methane gas production rate is the highest during the feeding. The period should at least cover about 50% substrate used and should not be short.

It has been realized that the sludge content and activity are subject to many changes during the reactor operation. After the stable operation, the activity will remain constant, but sludge content will increase steadily. The experiments for methanogenic sludge activity were carried out with glucose, and anaerobic biodegradability was performed with RCF-based kraft paper mill effluent and calculated successfully. The experiments were conducted with a 500 mL serum bottle with a septum. The test procedure is described below:

Anaerobic sludge 130 mL (3g VSS), glucose 4 g as feed, and nutrients $NaHCO_3$ (5 g), K_2HPO_4 (2g), and NH_4Cl (0.5 g) were mixed and made up to 1 L with tap water. The pH of the assay was maintained at 7.2. From this 1 L solution, 500 mL was taken in a serum bottle to calculate the methanogenic activity. The air in the headspace was replaced by Nitrogen gas. The bottle was placed in a water bath at 30^oC temperature, and the hose was connected to another bottle containing alkaline (KOH solution). The alkaline solution bottle was connected to a measuring cylinder to record the level of liquid displaced by biogas generated during the experiment. The experimental setup is the same for biodegradability as sludge activity, except wastewater (COD) is taken as feed instead of glucose, and nutrients are added as follows, $NaHCO_3$, g (amount of COD added g × L), K_2HPO_4, mg (amount of COD added g × 54) and NH_4Cl, mg (amount of COD added g × 16). The experimental setup is shown below (Fig. 6).

RESULTS AND DISCUSSION

The effluent was analyzed as per the standard test method of BIS and APHA. The results of the analysis of effluent samples for various pollution parameters are given below (Table 2). The analysis results show that the mills operate at low freshwater consumption, and effluent generated has COD & BOD on the higher side. The higher COD and BOD show that effluent carried the more organic load and is good for bio methanation to produce biogas.

By characterization of anaerobic sludge, the sludge profile was determined. The results of the analysis are given below (Table 3). The results were encouraging, with 23 $g.L^{-1}$ VSS and 37.78% organic content.

The experiment performed to determine sludge activity with glucose was performed successfully, and calculations are shown below:

Maximum methane production rate (l/day) or y/x (Fig. 7) = (0.630–0.480)/28 – 20 = 0.02

Amount of sludge (g VSS) = 1.5

$$\text{Sludge activity } (\text{g COD.gVSS}^{-1} \times \text{day}) = \frac{y/x \times 24 \times 2.6}{\text{Amount of sludge (in g VSS)}}$$

$$= 0.02 \times 24 \times 2.6/1.5 = 0.832$$

The cumulative gas production to calculate the y/x value is in Fig. 7. The results were satisfactory. The methanogenic activity of sludge was found to be 0.832 $gCOD.gVSS^{-1}\times Day$. This shows that the microbes present in sludge were healthy and active.

SEM Study of Granules

Visual examination of granular biomass shows a spherical shape and black. On the surface of granules, some irregular projects were also seen. SEM images of granules showed that the surface of granules was rough and uneven. The size of granules was 1 to 5 µm and exhibited a good settling tendency. The morphology of the granules demonstrated

Table 1: Sludge and substrate concentration for methanogenic activity assay.

S. No.	Experimental Set-Up	Sludge g $VSS.L^{-1}$	VFA/Sucrose g $COD.L^{-1}$
1.	Stirred	2.0 – 5.0	2.0 – 4.0
2.	Unstirred	1.0 – 1.5	3.5 – 4.5

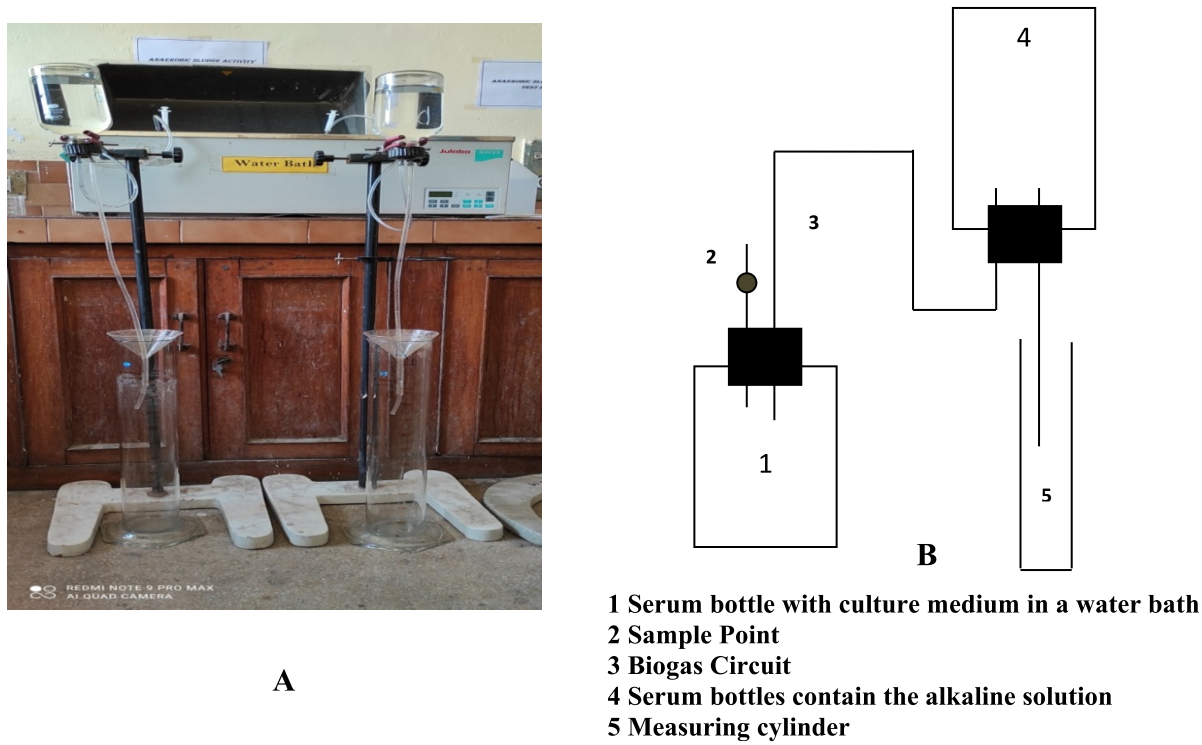

Fig. 6: Experimental setup with serum bottle to determine anaerobic sludge activity & biodegradability (A), Flow diagram with details (B).

the presence of a heterogeneous bacterial population on the surface of the granules (Fig. 8).

The biodegradability results of effluent are shown below:

The total amount of COD (COD_x) added to the bottle was = 2 g

The amount of gas produced by a bottle containing effluent was (Gas_x) =0.66 & 0.68 L

The amount of gas produced by blank bottles containing sludge & tap water was (Gas_B) = 0.12

$$\text{Biodegradability } (\%) = \frac{(Gas_x - Gas_B)}{COD_x} \times 260$$

$$= (0.66-0.12) \times 260/ 2 = 70.2$$

$$= (0.68-0.12) \times 260/ 2 = 72.8$$

The average biodegradability (%) was found to be about 71.5. The results show that the effluent collected from RCF-based kraft paper mills contains a high organic load, easily biodegradable with anaerobic bacteria by anaerobic digestion.

Table 2: Analysis results of the effluent of RCF-based kraft paper mills.

S. No.	Parameters	Method Followed	Mill – A	Mill – B	Mill – C
1.	pH	IS: 3025; P- 11	6.37	6.27	6.87
2.	TS, $mg.L^{-1}$	IS: 3025; P- 15	14,873	13,481	13,677
3.	TSS, $mg.L^{-1}$	IS: 3025; P- 16	3,171	2,786	3,688
4.	TDS, $mg.L^{-1}$	IS: 3025; P- 17	10,862	10,389	9,664
5.	COD (As Such), $mg.L^{-1}$	IS: 3025; P- 58	8,412	8,058	7,596
6.	COD (Soluble), $mg.L^{-1}$	IS: 3025; P- 58	8,028	7,639	7,073
7.	BOD (As Such), $mg.L^{-1}$	IS: 3025; P- 44	3,216	3,029	2,158
8.	BOD (Soluble), $mg.L^{-1}$	IS: 3025; P- 44	3,052	2,844	2,019
9.	Inorganic Solids, %	IS: 3025; P- 18	52.35	55.81	57.36
10.	Organic Solids, %	IS: 3025; P- 18	47.65	44.19	42.64

Table 3: Sludge profile results.

S. No.	Parameters	Unit	Method Followed	Results
1.	Total Solids (TS)	$g.L^{-1}$	IS: 3025; P- 15	61.83
2.	Suspended Solids (SS)	$g.L^{-1}$	IS: 3025; P- 16	60.00
3.	Volatile Suspended Solids (VSS)	$g.L^{-1}$	IS: 3025; P- 18	23.00
4.	Inorganic Solids	%	IS: 3025; P- 18	62.22
5.	Organic Solids	%	IS: 3025; P- 18	37.78

CONCLUSIONS

The findings of the mill visit indicate that the RCF-based kraft paper mills increased the reuse & recycling of backwater into the process, which extremely reduced their freshwater consumption & wastewater discharge. The characterization results of effluent collected show a high organic load in the effluent and have good potential to generate methane by anaerobic digestion. Sludge profile & activity results show that the anaerobic sludge used in the experiment was healthy & active. The methanogenic activity was found at about 0.832 $gCOD.gVSS^{-1}$×day, which proves the above statement. The visual examination of granular sludge with SEM also confirms the presence of anaerobic biomass. The biodegradability results were encouraging, with an average biodegradability of 71.5%. It indicates the effluent contains organic load, which can be easily treated with anaerobic digestion. The process generates methane, a value-added product from wastewater that can be used as an energy source.

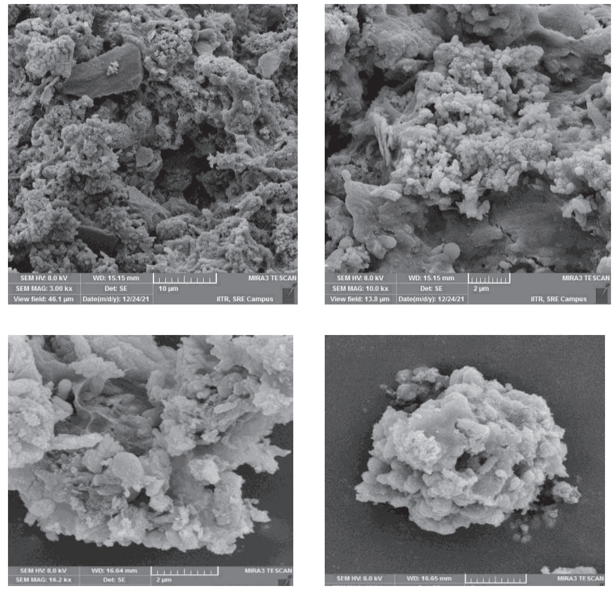

Fig. 8: SEM images of the heterogeneous bacterial population on the surface of the granules.

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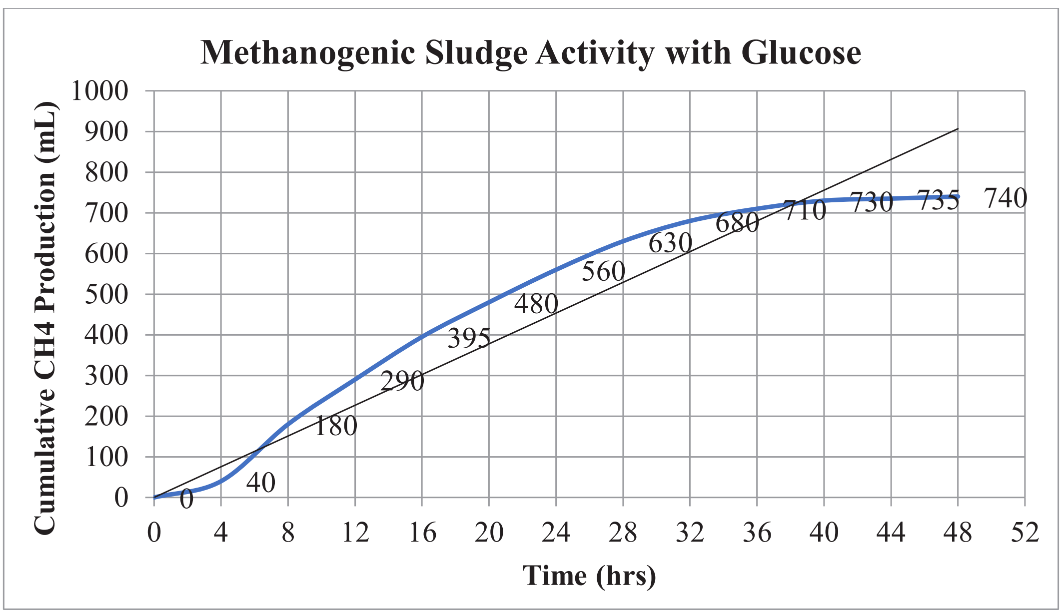

Fig. 7: Cumulative methane production from glucose.

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