

# Energy Recovery from Textile Mill Sludge Through Biomethanation

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## ABSTRACT

In the present study, anaerobic treatment of textile mill sludge was carried out. The detailed composition of solid textile mill sludge is necessary to ascertain feasibility and to select a suitable process for treatment and resource recovery. The waste brought from textile effluent treatment plant of local industry was assessed for various parameters. The present study was undertaken to explore the possibility of use of textile mill sludge as a substrate for biomethanation. Gas production was directly proportional to pH. As the pH increases from 6.3 to 7.4 gas production was increased. The methanogenic activity is sensitive to pH, so pH should be maintained neutral. The TVFA content in the digester was also affected by pH and was found to be inversely proportional to TVFA. The complete digestion resulted in total COD reduction by 63%.

**Keywords:** Energy recovery; textile mill sludge; biomethanation, anaerobic digestion; COD, pH; FVA, India.

## 1. INTRODUCTION

There are approximately more than 2500 textile processing units in India out of which more than 700 units are integrated textile mills with gross annual production of 9000 million meters of different variety of cloth (Bal, 1991). Their effluents constitute a major part of the total industrial effluents in India. The textile industry is not a single entity but encompasses a range of industrial units, which use a wide variety of natural and synthetic fibres to produce various fabrics. The treatment of the textile wastewater results in the formation of two end products: (i) the treated

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wastewater, and (ii) the sludge. The sludge can be stabilized by aerobic and anaerobic process. Aerobic process is not cost effective to handle large volumes. Anaerobic process has many advantages over the other process of organic waste treatment. The sludge is stabilized by anaerobic process also known as Biomethanation for energy recovery and organic waste treatment. It is a technologically simple method with the twin advantage of energy production and environmental conservation. The energy generated in the form of methane when utilized efficiently not only improves the overall economy but also provide on-site solution to waste management problem. So experiments were conducted on potential of biogas generation from textile mill sludge.

## 2. MATERIALS AND METHODS

The solid textile mill sludge was collected from H.P. Textile mills Ltd., Hisar (India). The constituent classes of organic matter, i.e. , volatile fatty acids were analyzed. The start-up period of an anaerobic film reactor is directly proportional to the concentration of the microbial population. Rate of start-up depends on the type of inoculum, the type and strength of waste, level of volatile acid maintained and the characteristic of the support material used. Two aspiratory bottles of 5L were connected with rubber tube. One of these was used as digester (No. 1) and another as the gas collection bottle (No. 2). Water displaced from digester (No. 2) by the gas produced in the digester (No. 1) was collected in the third bottle. Provision was made for feeding from the top of the digester and dilution was made from bottom. The feeding inlet and outlet tubes were kept closed with pinch cork and opened when required. The whole assembly was placed in an incubator at 37°C.

In the start of experiment, one kg textile waste (250 gm waste + 750 ml distilled water), one litre biogas plant slurry was used as inoculum. It was added in the digester (No. 1) and the volume was finally made up to 5 litres by adding distilled water. The temperature of BOD incubator was set at 37°C throughout the study. Daily 250 ml slurry from digester was taken to analyze the pH, COD, TVFA for 60 days. Addition of 250 ml of wastes (62.5 gm of sludge, 187.5 ml of distilled water) was made everyday. The hydraulic retention time in the digester was 20 days.

## 3. RESULTS AND DISCUSSION

Physico-chemical composition of textile effluent collected from H.P. Textile mills Ltd., Hisar (India) has been encapsulated in Table 1. The pH of the sludge was 8.4, which is in alkaline range. The water content of the sludge was very high even after dewatering it during the treatment process. It contained about 64-65% moisture in it. The chemical oxidation demand (COD), a measure of oxidizable matter and indirectly the organic matter in the sludge, was 64000 mg/L. The total solid was 197 g kg<sup>-1</sup>. The Total organic carbon (TOC) was very high (142 g/kg) and Total Kjeldhal Nitrogen (TKN) was very low (0.75%). So the C: N ratio was also high 199. The constituent classes of organic matter i.e. volatile fatty acids were analyzed. It is a direct substrate for microbial growth and metabolism and represents about 30-35% of organic matter in the sludge.

The nutrients and heavy metals were analyzed in the sludge by digesting it with diacid mixture. There was only small proportion of phosphate sufficient as nutrient but not a buffer system. The amount of potassium was 0.09 g kg<sup>-1</sup>. All the micronutrients analyzed were present in trace amount (0.04 to 2.5 g kg<sup>-1</sup>) except iron which was about 18.1–19.6 g kg<sup>-1</sup> (Table 2). The heavy

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metal content was analyzed using Atomic Absorption Spectrophotometer (AAS) in the diacid digest. Copper, Nickel, Chromium and Lead were present in a considerably higher proportion. These metal ions are required as micro-nutrients in methanogenesis. They increase the rate of methanogenesis both in thermophilic and mesophilic range of temperature. Iron, copper, nickel, cobalt, molybdenum, metal ions are known to accelerate methanogens.

### **Gas Production**

Daily gas production was monitored by the volume displacement in a graduated aspirator bottle of 5L capacity. The mesophilic anaerobic digestion result in production of biogas which consists of methane, carbon dioxide, and traces of H<sub>2</sub>. The potential for methane production depends on the status, type and constituents of the organic materials undergoing fermentation and these affect the quality and quantity of biogas (El-Shinnawi et al., 1989). Deivanai and Bai (1995) suggested that a higher content of cellulose (42.0%) and a lower content of lignin (14.0%) in banana trash are suitable potential feed material for biogas production.

The daily profile of gas production showed a sudden decline after two days of reactor inoculation with a minimum of 97 mg/L/day of gas formed on 7th day (Fig. 1). This could be due to fast depletion of the substrate in the reactor and a decrease in pH from 8.4 to 6.3 lead to accumulation of VFA as described earlier. It was also observed that maximum gas production was observed on 7th day onward till day 18, during this period there was no fall in pH which indicates an active stage of acidogenic and methanogenic phase. From 19th day onward there is decrease in gas production till 28 day, it is due to the decrease in pH 6.5 – 6.2. Thereafter the gas production ranged between 160±10 – 180±10 mg L<sup>-1</sup> day<sup>-1</sup> till 54th day. After 54th day there was a fall in gas production, which could be due to scarcity of the substrate in the digester. Thus the biodegradable fraction of the waste seems to be completely digested by day 53, beyond which only the non-biodegradable fraction could be present and hence there is no substrate available for conversion to volatile fatty acid. To enhance the activity of methanogenic bacteria different substrate could be used. Biogas production could be enhanced from cattle dung supplemented with 10, 20 and 30% poultry waste for gas production up to a concentration of 30% (Kanwar, and Guleri, 1994).

### **Variation in pH**

The pH of a digester is a measure of its acidic or alkaline content and is controlled by a series of physico-chemical equilibrium involving gaseous CO<sub>2</sub>, soluble CO<sub>2</sub>, bicarbonate ion (HCO<sub>3</sub><sup>-</sup>), ammonium ion (NH<sub>4</sub><sup>+</sup>) and the volatile acids. The primary effect of pH on digestion is to alter the rate of reaction of methane formation. The methanogens present in anaerobic digestion process are greatly affected by even small pH change, while the acid producing (acidogenic bacteria) can function satisfactorily at a wide variety of pH value. The effective pH range for methane production is approximately 6.5 to 7.5 with an optimum range of 6.8 to 7.2. Maintenance of this optimum range is important to ensure efficient gas production and to eliminate digester upsets.

The pH of digester was monitored daily before feeding fresh sample. To maintain the pH neutral 1M NaOH was used. This decrease was however, temporary, once the methanogens become active; the pH remained more towards the neutral (Fig. 1). Magbanua et al. (2001) have also reported that the pH initially 5.0 ± 0.5 for the hog waste and 6.0 ± 0.5 for the poultry dropped over first 15-20 days to as low as 4.5 ± 0.5, but eventually stabilized as 6.0 ± 0.5 in hog waste and at 7.0 ± 0.5 – 7.5 ± 0.5 in poultry waste.

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There was a sudden fall in pH from 8.4 to 6.3 within 5 days of reactor incubation. This in turn cause a sudden fall in gas production rate, which is minimum amount of gas produced during 60 days (97 mg/L/day) (Fig. 1). The sudden fall in pH from 8.4 to 6.3 could be due to faster depletion of the substrate in the reactor or accumulation of volatile fatty acids in the digester (Fig. 2). The pH started increasing on sixth day onward and attained the level of 7.6 on 12 day. During this period there is no fall in pH, rather there is a gradual increase in pH which indicates that acidogenic and methanogenic phase is co-existing and hence there is no accumulation of volatile fatty acids. To maintain the optimum pH dilute H<sub>2</sub>SO<sub>4</sub> was added to decrease the pH. From 13-day onward the pH started to decrease and reached at  $6.2 \pm 0.5$  on 23-25 day. The reported optimum pH range for methane producing bacteria is 6.4 – 7.2 whereas it is 5.5 for acid producing bacteria (Fongsatitkul et al., 1995). The petrochemical wastewater used for phase separation studies was suitable for biomethanation, because of its acidic nature (Patel and Madamwar, 2001). During the entire study period 6.2 was the minimum pH, which was attained between 23-26 days. From day 26 to 46, the pH varied between 6.3-6.9. Afterward it remained somewhat constant the range of 7.0 to 6.9 in the remaining days of experiment.

### **Total Volatile Fatty Acids (TVFA)**

The concentration of volatile fatty acid was used as an indication of activity of acidogens. Once the methane started forming concentration of acids start decreasing. A sudden fall in pH from 8.4 to 6.3 after 5th day of inoculation could be due to accumulation of volatile fatty acids in the digester (Fig. 2). From 5th day onward till 19th day the TVFA content of digester ranged between 45-70 mg/L. Maximum accumulation of volatile fatty acid was observed between 20-26 day i.e. 73-90 mg/L. From 28th day onward the TVFA content showed no significant difference with a value of  $75 \pm 10$  mg/L till 54th day, afterwards there is a gradual decrease in VFA on termination of experiment. As shown in Fig 2, from 5th day onward till 16th day there was a gradual increase in pH which indicated that acidogenic and methanogenic phases were co-existing and hence there was lesser accumulation of TVFA during this period (between  $45 \pm 10$  to  $55 \pm 10$  mg/L) (Rajeshwari et al., 1999). The increase in pH (acidic in nature) in turn increases the activity of acidogenic bacteria causing more degradation of complex organic matter into volatile fatty acid. The presence of volatile fatty acids indicates that all the acids were not utilized by methanogens.

### **Chemical Oxygen Demand (COD)**

The COD of the sludge mixture was monitored during the course of digestion to check the digestion level of the sludge. It is a measure of oxidizable matter and indirectly the organic matter present in the sludge. The organic loading in the digester has the COD of 16 g/day. The initial COD of the sludge was very high i.e. about 64000 mg/L, so the sludge was diluted four times to make the COD approximately 16000 mg/L. The inoculum in the form of biogas plant slurry was added to the digestion mixture, so as to give a boost to the micro-organism for methane formation. Nandy and Kaul (2001) have reported that for pharmaceutical wastewater start-up behaviour depicting seeding and maturation characteristics of the reactor system with respect to substrate removal efficiency as a function of time until the reactor system is fed with the wastewater.

During the digestion phase extending up to 29-30th day, there was 29% reduction in COD (Fig 3), and at day 47 the reduction in COD was 62%. However, further reduction in COD was 60-63%. As the reduction in COD becomes constant, it can be concluded that the substrate available

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to microorganisms for degradation is exhausted. Keeping these results in view the digester was dismantled after 60 days (Fig. 3). The complete digestion resulted in a total COD reduction of 63% comparable to that obtained for synthetic and thermo-mechanical pulping (TMD) wastewater with a loading rate of 15 kg COD m<sup>-3</sup> d<sup>-1</sup> and 50-60% COD removal efficiencies were achieved in 50 days in thermophilic UASB reactor (Rintala, 1991).

#### 4. CONCLUSIONS

The textile effluents are extremely diverse, and perhaps, one of the most potential pollutants to our aquatic environment. From the study it is concluded that sludge of a textile industry could be used as a potential source of energy by production of biogas. This gas could be used for various industrial as well as domestic purposes. Gas production is directly proportional to pH. As the pH increase from 6.3 to 7.4 results increase in gas production. The methanogenic activity is sensitive to pH, so pH should be maintained neutral. The TVFA content in the digester was also affected by pH and is found to be inversely proportional to TVFA.

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Table 1. Analytical parameter of the textile mill sludge

Analytical parameter	Sludge
PH	8.4
Moisture (%)	64
Total solid (g kg <sup>-1</sup> )	197
COD (mg/L)	64000
TOC (g/kg <sup>-1</sup> )	142
Nitrogen (%)	0.74
C:N ratio	199

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Table 2. The macro-micro-nutrient composition of textile mill sludge

Nutrients	Concentration (g/kg)
Phosphorus	0.04
Zinc	72.40
Manganese	68.56
Copper	96.48
Nickel	54.36
Chromium	22.84
Cadmium	3.60
Lead	34.46
Aluminium	232.80
Iron	18.1-19.6
Potassium	0.09

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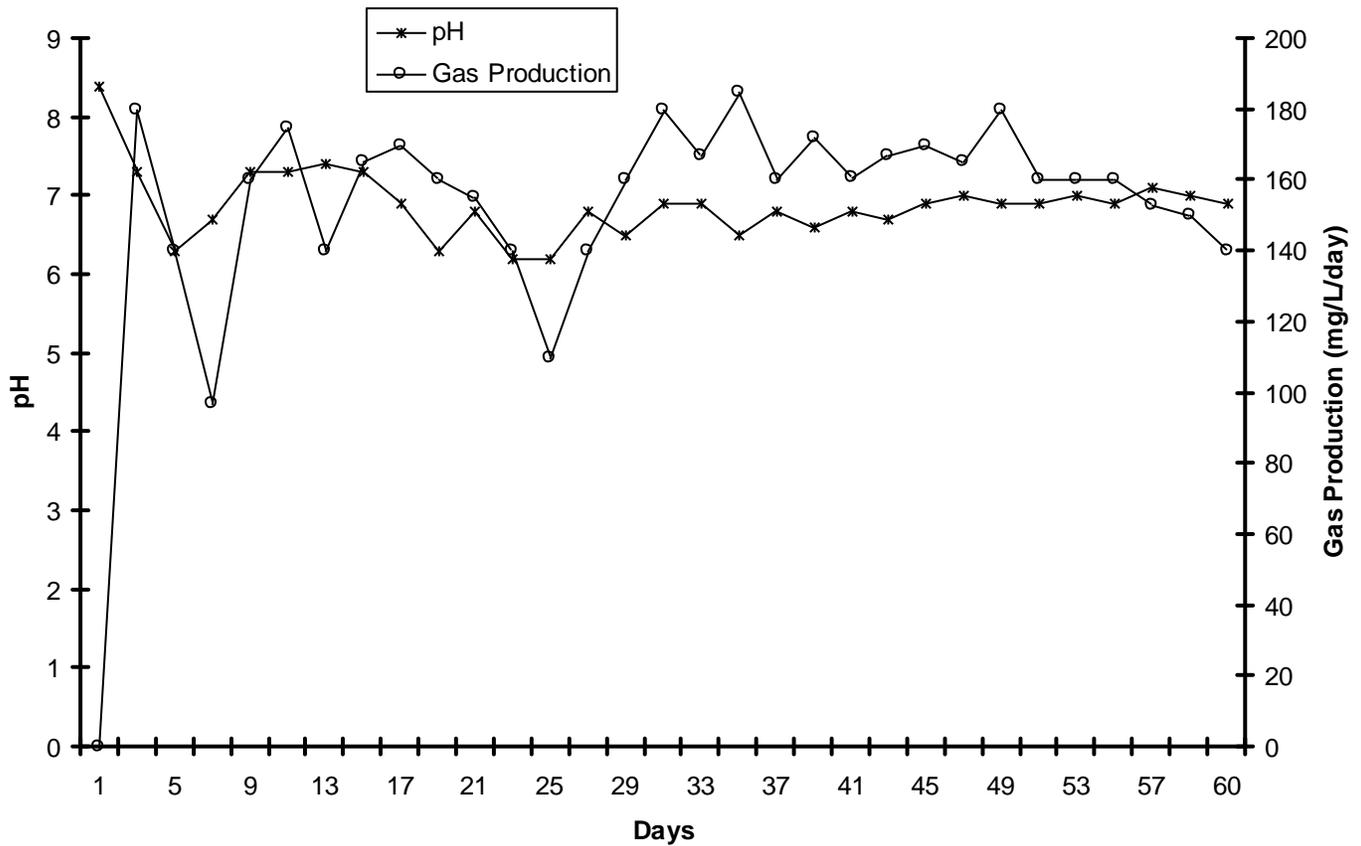


Fig. 1. Variation in pH and gas production during digestion

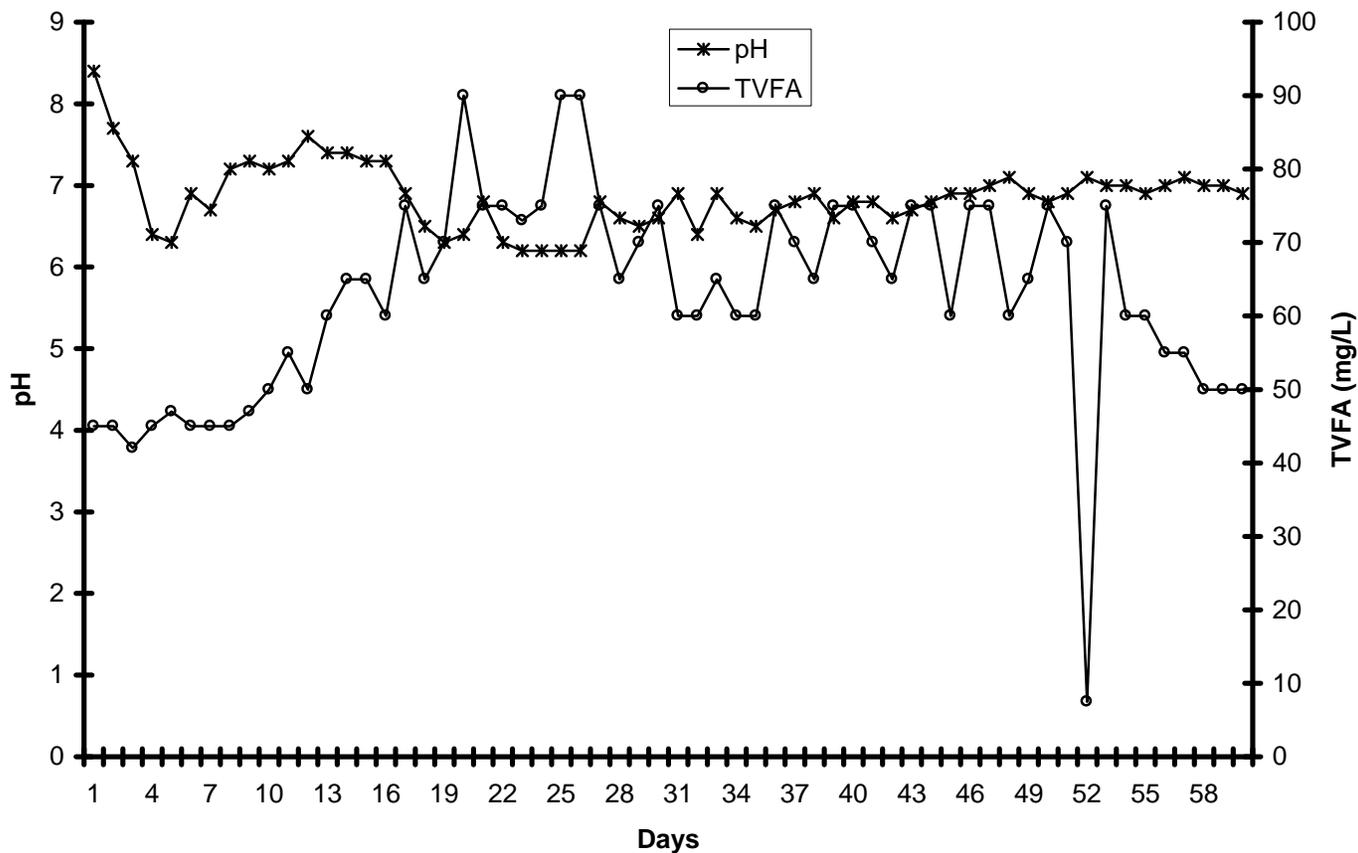


Fig 2. Variation in pH and TVFA during digestion period

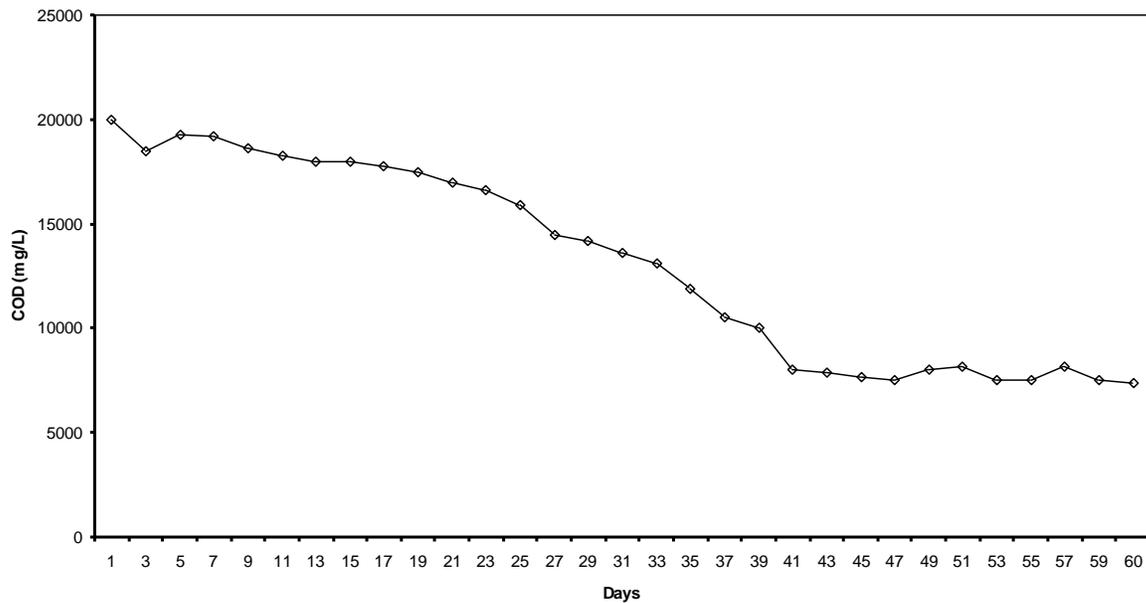


Fig 3. Pattern of COD variation of the sludge during the study period