# A bio-kinetic study on liquid-to-biogas transfer in an up-flow anaerobic sludge fixed film reactor in the treatment of sago wastewater

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#### ABSTRACT

Sago wastewater having intolerable odor with higher chemical oxygen demand (COD) leads to environmental damage. When it is treated appropriately, wastewater may possibly be utilized for irrigation and aquaculture. The present study reveals the biogas generation during the treatment of real-time sago wastewater using a laboratory-scale up-flow anaerobic sludge fixed film reactor with a working volume of 28.07 L. The reactor attained the steady-state on 14<sup>th</sup> day itself due to the combination of up-flow anaerobic sludge blanket reactor and fixed film process in a single reactor. The results indicated that the maximum biogas was attained at 0.151 m<sup>3</sup>/kg COD with the maximum COD removal efficiency being 92.076% at an organic loading rate of 0.119 kg COD/m<sup>3</sup>d with 4.5 d hydraulic retention time.

Keywords: Biogas; Chemical oxidation demand; Effluent

### 1. Introduction

The chief producers of tapioca in India are Kerala, Tamil Nadu, and Andhra Pradesh. These three states produce about seven million tonnes of tapioca every year. An approximate of 200 kg of sago is yielded from per tonne of processed tapioca roots. Being a water-consuming crop, it takes around 8,000 L of water to achieve the yield, that is, one ton of tapioca. In the production of one-tonne sago from tapioca, close to 30,000 to 40,000 L of effluent gets generated. The effluents are organic with high polluting effects on the environment. Tamil Nadu has a dense population who are involved in the cultivation and processing of tapioca and sago, thanks to the stringent rules put forth by the then British government during the Second World War that prevented the imports of foreign sago and starch from Singapore, Malaysia, Holland, Japan, and the USA. There seems to be a tremendous growth observed in sago and tapioca starch industry at Salem district, Tamil Nadu for the past four decades. Industries like sugar, sago (tapioca-based starch), and dairy are known for generating huge amounts of biodegradable waste streams. Such industries require water in huge quantities for its utilities such as boiler and other grey areas like washing, cleaning, etc., anaerobic treatment for wastewater has attained cult status when compared to aerobic treatment. Comparatively, the anaerobic treatment for wastewater is cost-effective, produces low sludge whereas it produces a high amount of biogas. Various researchers assessed the performance of this treatment earlier [1]. Effluent treatment via thermophilic systems with several phases would afford elevated treatment efficiency particularly with a stable progression [2]. The reactors with high volume would be appropriate for the treatment of industrial effluent [3]. The per capita water utilization turned out to be wastewater was about 60%–90% [4]. The 'up-flow anaerobic sludge blanket reactor' is one of the best promising candidates for anaerobic processes among the available ones, since it can maintain a high volume of biomass in the form of granulated sludge and it also can

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yield high-rate reactor performance [5]. A major drawback in this up-flow anaerobic sludge blanket reactor (UASBR) is that it consumes too much time, that is, several months for the granulation process. So, it requires modifications in the UASB process to overcome the drawback [6].

In order to get rid of this problem, some internal packing is used to maintain the biomass in the UASBR. The packing medium which is present in the reactor helps to increase the solid retention time [7]. The UASBR has been successfully used for treating various kinds of industrial wastewater [8]. The performance of Hybrid UASBR in the treatment of low-strength bilge water was investigated and a maximum chemical oxygen demand (COD) removal efficiency of 75% was achieved in the study [9]. The up-flow anaerobic sludge fixed film (UASFF) was a hybrid reactor with a combination of an up-flow anaerobic sludge blanket and an up-flow anaerobic fixed film reactor [10].

The process of gathering the biomass (cells of microorganisms) and the retention in biomass systems is increased when attached with a fixed medium. In the fixed film process, microorganisms are immobilized on a support surface thus forming biofilms [11]. The application of areca fiber as a fixed bed to treat domestic wastewater with 89% removal of COD was attained [12]. The stability of the attached growth process is high when compared to the suspended growth process at the instance when wastewater has considerable changes in flow rate and concentrations. 84% COD removal efficiency with 3 d hydraulic retention time (HRT) was achieved by operating the fixed film reactor to treat the diluted high strength dairy wastewater [13]. The utilization of fixed films for the wastewater treatment process has been increasingly considered due to its inherent advantages over suspended growth systems such as simplicity of construction and elimination of mechanical mixing [14].

This research work has the following objectives

- Evaluation of the characteristics of sago effluent.
- Evaluation of the process parameter COD with respect to
  Organic loading rate (OLR)
  - Hydraulic retention time (HRT)
- Evaluation of the performance of UASFF for treating the sago effluent with respect to biogas production.

## 2. Experimental methodology

## 2.1. Hybrid reactor configuration

UASFF is a hybrid anaerobic reactor which is a result of combining UASB and up-flow fixed film reactors. In the UASFF reactor, the basal portion where the granular sludge and flocculants are produced is UASB whereas the fixed film portion above the UASB portion denotes the other one where the internal packing occurs. The researchers constructed a laboratory-scale UASFF reactor using a Plexiglass column of 15 cm internal diameter and 180 cm height. The reactor had a total volume of 0.03038 m<sup>3</sup> in which 0.02826 m<sup>3</sup> was the working volume, that is, without headspace. There were three compartments in the column such as the bottom, middle, and the top in which the height of the foremost part of 111 cm was operated as a fixed film reactor. While the middle section was packed with a packing medium, the top portion of the bioreactor functioned as a gas-liquid-solid separator. The top section, discussed above, has a specific function, that is, permit the segregation of biogas and washed out solids apart from the liquid phase. The biogas was passed on to the gas collector tank using an inverted funnel-shaped gas separator. In order to determine the composition of biogas, a gas sampling port was provided. The feed rate was controlled by the variable speed peristaltic pump (PP-30). Fig. 1 shows the schematic diagram of the laboratory-scale experimental setup. Configuration of the UASFF reactor is presented in Table 1.

## 2.2. Packing medium

Fujino spirals sized 26 mm were used as packing medium in the fixed film portion of the UASFF reactor. The packing material had a specific surface area, that is, 500 m<sup>2</sup>/m<sup>3</sup> with 87% void space. Fig. 2 shows the structure of Fujino spirals.

#### 2.3. Operation and monitoring

The required amount of sludge waste and sewage wastewater is collected from the nearby sump which is located at the Faculty of Agriculture, Annamalai University, Annamalai Nagar for the formation of inoculum in the fixed film. After stabilization, the sago industry wastewater is to be added with domestic wastewater in the reactor. The sago wastewater was collected from N.S Starch and Sago factory in Salem. Both influent as well as effluent samples were collected by the researchers every 24 h and were assessed immediately in alignment with the procedure put forth by APHA standard guidelines 2016.

#### 2.4. Determination of pH

The buffer solution of pH 4.02 and 9.14 at 300°C was prepared by dissolving the respective buffer tablets separately in 100 ml of fresh distilled water in a container. The temperature of the buffer solution has measured and adjusted to the temperature compensating control to the value of the temperature of the buffer solution, that is, 30°C. The container having the buffer solution of pH 4.02 was kept on the base plate of the electrode stand. The electrode holding clamp has clipped at the appropriate height on the rod of the electrode stand such that the electrodes were immersed in the buffer solution. The pH meter is standardized. Similarly, pH values were measured for the given samples. Reading were noted and tabulated.

#### 2.5. Determination of biochemical oxygen demand

Pure water was taken in a glass container and bubble compressed air for 2 d to attain saturation. 3 nos. of 1 L beaker with saturated water was taken. 1ml of manganese sulfate, potassium buffer solution, ferric chloride, and calcium chloride was added in each beaker and neutralized to pH 7. The first beaker is considered blank and in the remaining two beakers 2 ml of sample seed was added. Initial DO was founded out by titrating with 0.025N sodium thiosulphate solution using starch as an indicator. Then the burette reading was noted. After 5 d, the DO was determined in a similar way. DO of sample on 0<sup>th</sup> day, D1-DO of sample



Fig. 1. Schematic diagram of the laboratory-scale experimental setup.



Fig. 2. Fujino spirals.

on 5<sup>th</sup> day, CO-DO of sample on 0<sup>th</sup> day, C1-DO of sample on 5<sup>th</sup> day. Then, (biochemical oxygen demand – BOD)  $BOD = [[DO-D1]-[CO-C1]] \times dilution factor \times 100.$ 

## 2.6. Determination of COD

The sulphuric acid [0.2 ml] was added to the Reflux flask. The sample of [20 ml] was taken in the flask. The potassium dichromate [10 ml], silver sulfate sulphuric acid,  $H_2SO_4 - AgSO_4$  (30 ml) was added with it. The flask was connected to condenser and reflux for 2 h. The sample was made – up of 150 ml distilled water. Three drops of ferroin indicator were added with it. It was titrated against ferrous

Table 1 Configuration of the UASFF reactor

Reactor configuration	Dimensions	
Total height of the reactor, cm	180	
Effective height of the reactor, cm	168	
Effective volume of the reactor, L	28	
Effective diameter of the reactor, cm	15	
Diameter of the gas-liquid-solid-separator	3	
(GLSS) top, cm		
Diameter of the GLSS bottom, cm	12	
Total height of the GLSS, cm	10	
Diameter of influent and effluent pipe, cm	1	
Peristaltic pump	PP-30	

ammonium sulfate solution till the color change to green to wine red. The reading was taken by noting the burette reading.

## 3. Result and discussions

To investigate the performance of the UASFF reactor a laboratory-scale study was conducted for treating sago wastewater. The characteristics of sago wastewater is presented in Table 2. The reactor was started with a low initial loading rate for the successful start-up process and the principal parameters such as pH, COD, and biogas production are strictly observed. The start-up process of UASFF has been well presented in the literature [15].

Table 2		
Characteristics of	of sago waste	water

Sl. No.	Parameters	Sample (S1)	Sample (S2)	Sample (S3)	Desirable limit of IS 10500
1	рН	8.90	8.60	9.2	6.5-8.5
2	Total solids, mg/L	4,670	5,680	4,850	500
3	Total suspended solids, mg/L	1,600	1,550	1,650	100
4	Total dissolved solids, mg/L	3,070	4,130	3,200	500
7	BOD <sub>5</sub> @20°C, mg/L	1,750	1,206	1,658	30
8	COD, mg/L	4,400	3,880	4,160	250
9	Ammoniacal nitrogen, mg/L	73.00	85.60	84.20	50
10	Chlorides, mg/L	432	400	412	250
11	Turbidity (NTU), mg/L	14.8	16.60	12.50	1
12	Temperature, °C	28	29	32	<40
13	Sulfates, mg/L	225	205	218	200
14	Phosphate, mg/L	68.8	72.8	85	NA
15	Hardness, mg/L	800	1,200	950	200
16	Sodium, mg/L	833	900	710	200
17	Potassium, mg/L	524	685	490	NA
18	Calcium, mg/L	378	321	285	75
19	Lithium, mg/L	83.6	75.5	68.0	2.5

#### 3.1. Start-up process of the reactor

The reactor was a start-up with an initial OLR of 0.551 kg COD/m³/d followed by increased OLR and reduced HRT. The reactor was operated continuously until the steady-state condition was reached. The reactor was continuously fed with the initial influent 480 mg/L COD with 24 h HRT at the time of the start-up stage to being the process. The operating temperature varied from 25°C to 35°C (mesophilic range) which is more prominent. Also, the pH of the reactor was comparatively stable by varying from 6.8 to 8.3 which are well suited for methanogenic activities. This range of pH indicates that the reactor had sufficient alkalinity to neutralize the organic acids delivered from the hydrolysis as well as the acidogenesis stages. The start-up stage of the process was begun by continuous feeding of the reactor with an initial influent COD concentration of 480 mg/L with an HRT of 24 h and consequently OLR of 0.551 Kg COD/m<sup>3</sup>/d which is remarkably a low value. The COD removal rate during the first 2 d was low in the range of 30%–40%. The low efficiency in removal at the beginning of the process is due to the biomass adaptation in the new environment. The reactor achieved steady-state conditions during the period of 18<sup>th</sup> day to 21<sup>st</sup> day with a maximum COD removal efficiency.

#### 3.2. Acclimation and process stability

When the anaerobic reactor was started, the biomass was made to undergo acclimatization to suit the new environmental conditions such as reactor configuration, operating strategies, and substrate temperature. From the active biomass plant that is functioning at the Faculty of Agriculture, Annamalai University, Annamalai Nagar the inoculums for granular sludge were sourced. The granules were then screened to get rid of debris. The overall objective of the start-up process followed in the UASFF reactor is to establish the microbial consortium.

#### 3.3. Performance of the reactor after start-up

After attaining the steady-state condition in the start-up process the reactor was operated with increased OLR in order to achieve the most favorable loading rate of the reactor. If BOD/COD ratio of the sago wastewater is more than 0.6, the wastewaters are biologically treatable without acclimatization; and if the ratio is less than 0.6 and up to 0.3, then acclimatization is needed for biological treatment; and if the ratio is less than 0.3, biological treatment may not be necessary. For sago effluent, the BOD/COD ratio is 0.57; therefore the acclimatization is needed for biological treatment.

The acclimatization of sago effluent in various mixing proportions is given below:

- 1<sup>st</sup> day 80% domestic sewage + 20% sago effluent
- 2<sup>nd</sup> day 60% domestic sewage + 40% sago effluent
- 3<sup>rd</sup> day 40% domestic sewage + 60% sago effluent
- 4<sup>th</sup> day 20% domestic sewage + 80% sago effluent
- 5<sup>th</sup> day 100% sago effluent

From the reactor, both influent, as well as effluent samples for every 24 h, are analyzed immediately. At the initial stages, the influent wastewater feed was collected from the treatment facility, Annamalai University, Chidambaram. With the purpose of making the UASFF reactor a successful one, the initial loading rates were kept at a low level. This was followed because it enhances the growth of anaerobic active sludge, thanks to low COD organic loading that eventually resulted in the low amount of gas production and low wastewater up-flow velocity. Few researchers have discussed the start-up process of UASFF in detail. The performance of HRT and HRT with respect to percentage COD removal efficiency is presented in Figs. 3 and 4.

The performance of HRT and OLR with respect to the percentage of COD removal efficiency is presented in Figs. 3 and 4. At the beginning of the process, the removal efficiency was low due to the adaptation of biomass in the new environment. The reactor attained steady-state conditions during the 5<sup>th</sup> day to 14<sup>th</sup> day with 90% COD removal efficiency. It is difficult to retail the efficient number of favorable microbes in the system.

In the anaerobic reactors, pH remains the important factor that controls the digestion process. The methaneproducing microorganisms can survive in conditions with pH values in the range of 6.6 and 7.6 though it is a fact that the stability can be achieved when methane is produced in the pH range of 6.0 to 8.0, it is advisable to avoid the pH values less than 6.0 and more than 8.3 since in these ranges, the methane-forming microbes may be inhibited. However, the reactor's pH was maintained under stable conditions when it varied in the range of 6.8 to 8.3, a favorable condition for methanogenic activities. This pH range denotes that the reactor was equipped with sufficient alkalinity for the neutralization of organic acids delivered from the hydrolysis as well as the acidogenesis stages. The reactor attained a steady-state condition on the 14th day itself due to the combination of UASBR and fixed film processes in a single reactor. During the start-up process, the maximum COD removal efficiency, that is, 92.06% was attained with 4.5 d HRT and 9.995 kg COD/m<sup>3</sup> OLR. The biogas produced was



Fig. 3. Performance of HRT with respect to percentage COD removal efficiency.



Fig. 4. Performance of OLR with respect to percentage COD removal efficiency.

maximum when COD removed was 0.119 m<sup>3</sup>/kg COD in the influent COD of 4976 mg/L with 3 g glucose as co-substrate at 4.5 d HRT.

## 4. Conclusion

The study concludes that the UASFF, with high performance, was capable to handle the high organic load and can treat any type of high strength wastewater. The reactor attained steady-state conditions within 7-14 d from the start-up of the day. The wastewater sample from the sago industry was collected and characterized. The UASFF reactor was successfully utilized to achieve high COD removal efficiency within a short start-up period. It was necessary to add glucose, at a concentration ranging from 1 to 3 g/L, as an external carbon source to the sago wastewater. The minimum biogas was produced at 0.009 m3/kg COD with 36.25% COD removal efficiency in the absence of co-substrate with 0.5 d HRT and OLR of 1.42 kg COD/m<sup>3</sup>d. The minimum percentages of methane and carbon-dioxide were 38.46 and 43.68. The maximum biogas was attained at 0.151 m3/kg COD with the maximum COD removal efficiency being 92.076% at an OLR of 0.119 kg COD/m<sup>3</sup>d with 4.5 d HRT. Thus the Fujino spirals used as packing media in the UASFF reactor were capable of retaining high biomass concentration present in sago wastewater. The maximum percentages of biogas and carbon-dioxide were 72.37 and 12.73.

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