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# Synthesis of Laboratory Scale Hydrogen Fuel from Distillery Spent Wash with Xylose as Co-Substrate

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**Abstract:** *In recent aura, the potentiality of hydrogen as an alternative fuel is a proven fact. Due to the higher calorific value and eco-friendly nature, it has already gained considerable popularity in laboratory scale though the commercialization of the same faced quite a challenge due to lower extraction values. The present work incorporates production of commercial grade biohydrogen fuel from distillery spent wash activated with different dosage of raw xylose. The yield was also investigated under two retention periods such as 8 hr and 12 hr, but in both the scenario the dose of 20 g/l found to be most effective. A significant difference was also observed both in terms of hydrogen yield and chemical oxygen demand (COD) reduction against the retention time with 12 hr Hydraulic retention time (HRT) found to be associated with more satisfactory values in both the cases which are approx. 196 ml (39.2 ml/d) and 65% COD reduction respectively. Future this paper also comprises to identify the indigenous species responsible for the hydrogen production by Scanning Electron Microscope (SEM) analysis. It reveals o cocci- and rod-shaped organism in accumulated sludge.*

**Keywords:** *Xylose, Dark Fermentation, Optimum Dosage, Biohydrogen production, Co-Substrate*

## I. INTRODUCTION

Hydrogen is a promising future alternative energy because it is clean and renewable and generates no toxic by-products in its combustion. Biohydrogen is an attractive hydrogen source because its energy saving production process as compared with chemical processes. Anaerobic hydrogen-producing enrichment culture applications that produce hydrogen from organics have received considerable attention (Taguchi et al. 1995, Fang and Liu 2002). Fermentative hydrogen production has been shown to have great potential for development as a practical biohydrogen system (Levin et al. 2004). Anaerobic sewage sludge contains a variety of mixed microflora and is well used for efficient hydrogen production from organic wastes (Lin and Chang 1999, Zheng and Yu 2004, Chen et al. 2001). A large amount of organic waste is produced from agricultural, industrial and domestic processes. Converting organic wastes into valuable products such as hydrogen gas, refuse derived fuels, composts and solvents is sustainable. Glucose and xylose are produced at a concentration ratio of 55–65% to 35–45% during the saccharidification of some organic wastes like wood, paper, agricultural by-products or crops (Ahring et al. 1996, Lavarack et al. 2002). Fermentative hydrogen production from glucose using mixed microflora has been well studied (Fang and Liu 2002, Lin and Chang 1999, Zheng and Yu 2004, Morimoto et al. 2004). However, fermentative hydrogen production from xylose using mixed microflora has not been well reported. Fermentation from xylose has been reported for ethanol production and hydrogen production from xylose is only cited using pure cultures (Taguchi et al. 1995, Ahring et al. 1996, Toivari et al. 2001, Taguchi et al. 1994). The pH and substrate concentration are essential and important environmental factors affecting biological processes. A change in system pH or substrate concentration might result in decreased process efficiency. To what extent xylose-degrading natural anaerobic microflora can resist these two environmental factors remains unknown. Understanding the influence of these two factors on biohydrogen production is necessary for developing xylose-based hydrogen fermentation applications (Lin and Cheng 2006).

This paper focus to investigate the most pertinent xylose dosage for optimal hydrogen yield from distillery spent wash and also to interpret the impact of various retention periods over the production rate.

## II. MATERIALS AND METHODS

The production of next-generation commercial grade hydrogen fuel from xylose using anaerobic dark fermentation includes some steps described as follows.

### A. Substrate Dosing

Xylose was used as a substrate for the extraction of bio-hydrogen. The experiment includes different concentration of dosing varying with different retention periods. A concentration ranging viz., 10 mg/l, 20 mg/l, 30 mg/l was used to conduct the experimental study. The influence of most appropriate dosing and its associated values for the other monitoring parameters were tabulated in Table 1.

Table 1: Influence of optimum dosing of 20 mg/l optimal HRT of 8 hr over monitoring parameters

Hours	H <sub>2</sub> in ml	VFA (mg/l)	COD removal (%)	Alkalinity (mg/l)	VFA/ Alk	Influent pH	Effluent pH
8	23	3266.5	12	4536.86	0.72	5.6	6.8
16	47	3158.2	19	4210.93	0.75	5.5	6.7
24	69	2954.2	26	4160.84	0.71	5.6	6.5
32	93	2932.6	34	4250.14	0.69	5.5	6.6
40	115	2870.0	41	4100.00	0.70	5.8	6.5
48	139	2793.4	46	3778.88	0.72	5.6	6.3
56	156	2720.8	52	3835.29	0.68	5.7	6.3
64	167	2608.0	55	3778.19	0.72	5.5	6.3
72	172	2720.3	58	3942.05	0.68	5.5	6.4
80	175	2680.6	60	3884.92	0.69	5.8	6.4

### B. Optimization of the Retention Period

The study includes conduction of the batch study over two different HRT values 8 hr and 12 hr respectively. The yield associated with these two values was significantly commendable and 12 hr found to be desirable due to higher treatment and production efficiencies and the same has been tabulated in Table 2.

Table 2: Influence of optimum dosing of 20 mg/l and optimal HRT of 12 hr over monitoring parameters

Hours	H <sub>2</sub> in ml	VFA (mg/l)	COD removal (%)	Alkalinity (mg/l)	VFA/ Alk	Initial pH	Effluent pH
12	26	2678.4	13	4320	0.62	5.5	6.6
24	58	2566.5	21	4425	0.58	5.6	6.7
36	82	2354.0	28	4280	0.55	5.5	6.2
48	105	2685.0	32	4262	0.63	5.7	6.5
60	128	2223.6	40	4300	0.52	6.0	6.5
72	151	2221.5	51	4356	0.51	5.8	6.5
84	169	2204.0	58	4408	0.50	5.6	6.6
96	183	2105.2	60	4128	0.51	5.7	6.7
108	191	2165.0	63	3906	0.55	5.8	6.4
120	196	2256.8	65	4109	0.56	5.6	6.5

### C. SEM Analysis

SEM analyses were carried out with Philips XL-30S FEG / FEI Quanta250 FEG brand of SEM Scanning Electron Microscope at the Department of Manufacturing Engineering in Annamalai University. After coating each sample with gold/palladium using Magnetron Sputter Coating Instrument at the Department of Physics in Annamalai University. The SEM analysis image was zoomed with Magnification ranging from 600X and 1100X respectively for better understating and interpretation purpose.

### III. RESULTS AND DISCUSSION

Xylose was added as a co-substrate along with the distillery spent wash for greater production of the biohydrogen fuel (Lin and Cheng 2006). Different concentration of the activating agent was tried in the form of xylose and a value of 20 mg/l found to be most effective associated with the optimum hydrogen yield and maximum COD removal of 65% (Hawkes et al. 2002, Shin et al. 2004, Rajaharharasudhan and Venkatesh, 2018). Figure 1 presented the yields of hydrogen fuel associated with different trails for various concentrations of xylose.

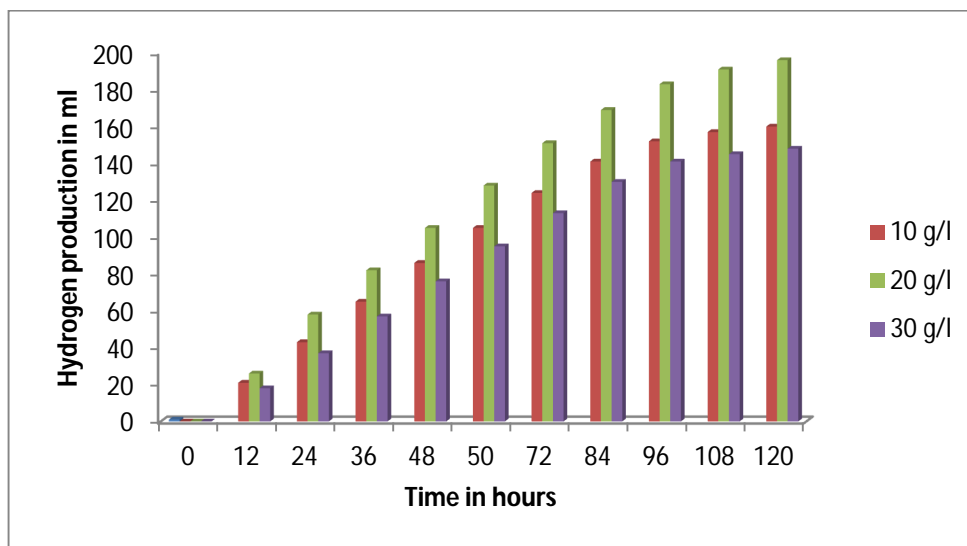


Figure 1: Variation in Hydrogen production Vs co-substrate concentrations at 12 hr HRT

As discussed earlier, the retention period and temperature also found to be playing a significant role in terms of optimization of the process parameters (Wu and Lin 2004, Chang et al. 2002). The present study includes two different retention periods and amid 12 hr resulted in potentially better values (Kongjan et al. 2009, Khamtib and Reungsang 2012). Though the figures associated with 8 hr retention time also need to be paid attention due to the stipulated treatment period and acceptable removal efficiencies (Dessi et al. 2017, Hu and Zhu 2017) explained in Figure 2.

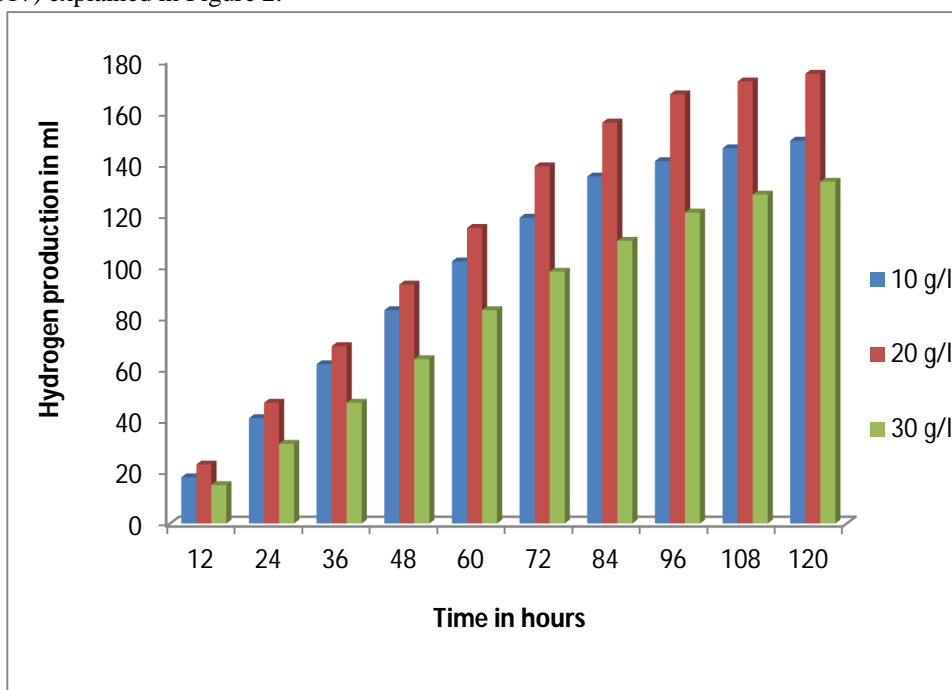


Figure 2: Variation in Hydrogen production Vs co-substrate concentrations at 8 hr HRT



Basic information on the nature and origin of particulate material can often be gathered from the size, shape and/or surface form of the individual particles. This type of information was readily obtained by examining the particles in SEM and the analysis report was enclosed in Figure 3 and 4 successively with different magnifications.

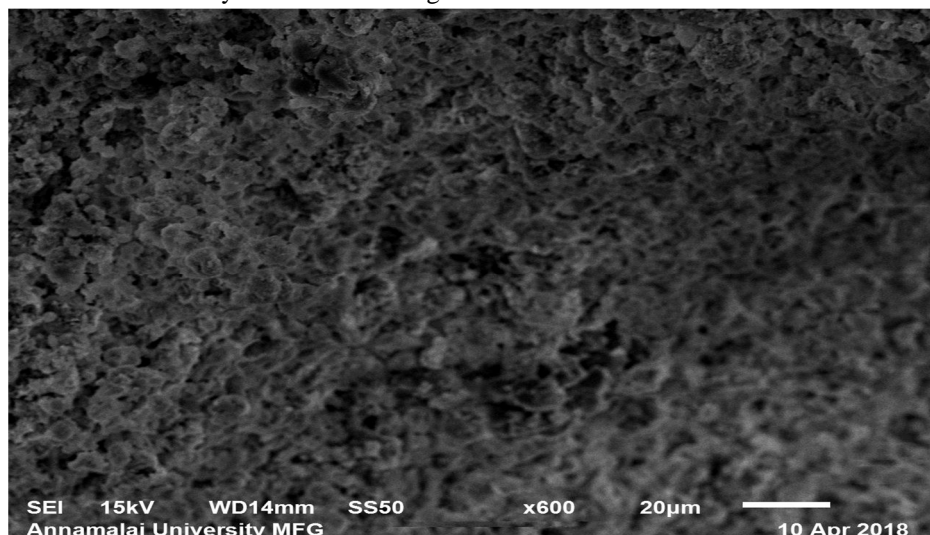


Figure 3: SEM Analysis imagery with Magnification ranging X600

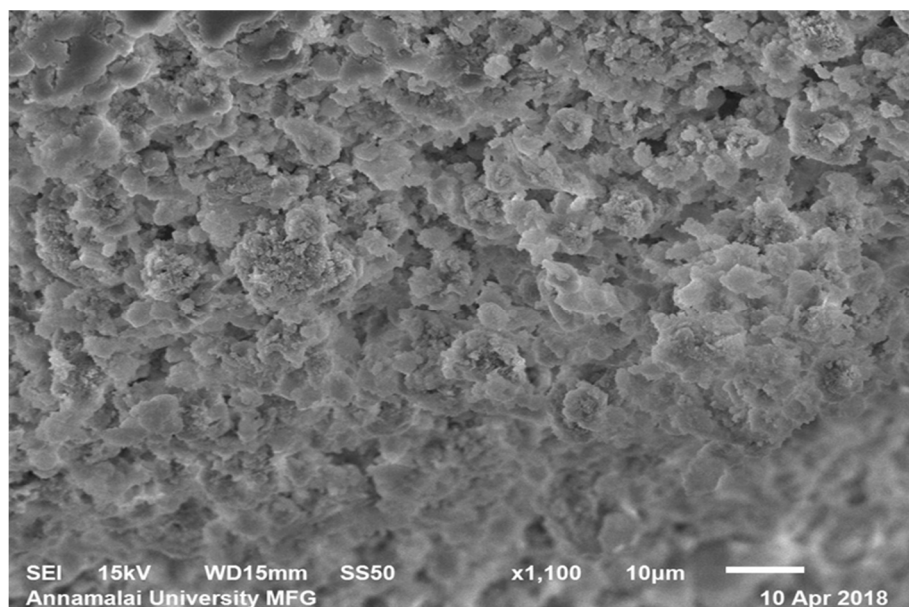


Figure 4: SEM Analysis imagery with Magnification ranging X1100

Visual examination of granular biomass revealed a black color with a spherical shape. Slight irregular projections were also seen on the surface of the granules. SEM photographs of the granules showed that the overall surface of granules was rough and uneven. Figure 3 and 4 shows the enlarged view of granules in a batch reactor. Hulshoff et al. (1986) have reported that the granules vary widely in shape depending on the operating conditions but they usually have a spherical shape. granules demonstrated the presence of heterogeneous bacterial population on the surface of the granules. Both filamentous and cocci shape microorganisms were predominant on the surface of the granules. The identification of the species was based on the morphology of the granules. Figure 4 shows a cluster of both cocci-shaped and rod-shaped microorganism dangling on the surface of granules. Shin et al. (1992) have also reported the predominance of both cocci- and rod-shaped organism in sludge acclimatized for anaerobic digestion of distillery-spent wash. Figure 3 shows the enlarged view of the cluster of cocci-shaped *Clostridium* type bacteria on the surface of granules. The high content of calcium, sulfur and phosphorous found in the sludge indicates the precipitation of these minerals inside the granules. This can be attributed to the presence of high content of calcium, phosphorous and sulfate in the neutralized spent wash

fed to the reactor (Pekguzel et al., 2015). Lin and Cheng (2005) has reported that since pH inside the granules remains higher than that of the bulk liquid, salts precipitation is more likely to take place inside the granules.

#### IV. CONCLUSION

The research concludes that the concentration of co-substrate plays a major role in optimizing the yield of biohydrogen fuel. On the other hand, the investigation also encountered a significant proportional correlation between the hydrogen yield and the COD and VFA reduction irrespective of the retention period. Thus, based on the above hypothesis it can be easily concluded that addition of co-substrate in the form of xylose definitely boosts the hydrogen production and it has a beneficial impact on hydrogen production. Ultimately, the research reports an optimal hydrogen yield of 196 ml (39.2 ml/d) with an associated COD removal fraction of 65% for 12 hr HRT.

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