

## Research article

### Influence of Abiotic Factors on Coir Retting Process

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Received: 26 March 2022, Revised: 12 May 2022, Accepted: 13 September 2022

DOI: 10.55003/cast.2022.02.23.013

#### Abstract

##### Keywords

coconut husk;  
volatile fatty acid;  
chemical oxygen demand;  
polyphenol;  
leachate;  
sludge;  
pectin;  
tannin

Coconut husk represents the entire fibrous envelop in the fruit and constitute both the exocarp and mesocarp in the raw material for the coir industry. Retting results in separation of this fibrous envelop which is an assemblage of fibers with cork like parenchymatous cells containing a cementing material dispersed throughout the mass. The present study attempted to determine the effect of pH, aeration and degradation capability of aerated leachate on coconut husk retting. The experiment was done in seven buckets of 5L capacity and the pH of water in each bucket was adjusted from 3.0-9.0 with 600g of 11 months old fresh coconut husks immersed in each bucket. The parameters of color, pH, volatile fatty acid (VFA), chemical oxygen demand (COD) and polyphenols were analyzed regularly during the study. The effects of aeration and the degradation capability of aerated leachate was also studied. The VFA and COD level increased initially and then decreased. Neutral pH was found to be ideal for the leaching process.

#### 1. Introduction

Kerala is proud to have the valuable gift of coir, or golden fiber, which is one of the oldest traditional industries in the state, and employs tens of thousands of people. Kerala's coir industry is one of the state's fastest-growing industries [1]. Coir is a natural fiber made from the husk (mesocarp) of the coconut, *Cocos nucifera* (L.), which belongs to the family Arecaceae. Lignin, tannin, cellulose, pectin, and other water-soluble components make up the fibers, which are usually 50-350 nm in length [2]. Coconut husk has a fibrous leathery coating that protects the seed from the mechanical shock of extended falling distances of up to 25 m and imparts water resistance. Long individual

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fibers are linked together with pectin cementing material by corky parenchymatous cells [3]. The pith is made up of spongy cork cells from the non-fibrous component of the mesocarp, which accounts for around half of the total weight of the husk, with the rest consisting of coir fiber, polyphenol, pectin compounds, and other chemicals. The husk fiber is extremely strong, consisting of a bundle of 30-300 micro fibrils. Coconut fibers were used to make coir ropes and coir products such as matting, rubberized coir materials, and a range of other related items [4]. It has additional benefits such as stronger hardness quality (free of brittle qualities such as glass), superior acoustic resistance, non-toxicity, moth resistance, resistance to bacterial and fungal degradation, and it has relatively low flammability [5, 6]. As opposed to synthetic materials, which have issues with biodegradability and recycling, and which are associated with health concerns, coir's 100 percent biodegradable nature has resulted in a surge in demand [7].

During the process of coconut retting, a large quantity of organic matter, which is both biodegradable and non-biodegradable and colloidal substances are released into the water, turning the water turbid and blackish. The phenols, aromatic hydrocarbons and their derivatives from the husk constantly leach out from the husk into the surroundings, causing extensive damage to aquatic flora and fauna [8]. Large quantities of toxic phenols and organic substances like pectin, pentosan, tannins and fat are discharged into water bodies during retting time. The retting units mostly discharge the effluent directly into the estuaries without any treatment. This leads to the deterioration of both the physical and biological conditions of the water. To overcome this situation, we need an efficient mechanism for the treatment of retting effluent, and a new way to recycle it into a usable form. Actually, retting by natural means is a lengthy process that normally takes 6-10 months. So many factors, both abiotic and biotic, influence the process of retting. Pretreatment of husk with strong acid and alkali leads to environmental pollution, so we have tried an ecofriendly approach for effective utilization of coir fiber. Coir retting is a natural process but a study of the impact of both biotic and abiotic factors at each stage of retting in order to find out the fastest way to complete the retting process was needed. Furthermore, we decided to look at a better way of reusing coconut husk leachate. Considering all of the above, this study focused on finding out the ideal pH, the influence of oxygen on the retting process, and the degradation capability of aerated leachate in an anaerobic system. Moreover, this study aimed to ret the coconut husk without accumulating any toxic hazardous products and to ret it in a short period.

## **2. Materials and Methods**

### **2.1 Sample –Coconut husk**

Eleven months old green mature raw coconut husks were selected for this study because maturation of coconut takes approximately 11 months. In immature coconut, the husk is not developed properly. In dry coconut, the husk being dehydrated results in delays of the retting process. The coconut husk that was collected from a single plot of cultivable land at Thiruvananthapuram, Kerala, India was used for this study. The chemicals used for this study were procured from Merck-India Ltd, Mumbai.

### **2.2 Experimental set up**

#### **2.2.1 Effect of pH on coconut husk retting**

The experiment was done in seven buckets of 5 L capacity. Each bucket was filled with 3 L of tap water, and the pH of the water in each bucket was adjusted from 3 to 9 using dilute 0.1 N NaOH and 0.1 N HCL. About 600 g of fresh coconut husk (11 months old) was immersed in the water in

each bucket. The age of the husk was important in chemical composition and leaching properties. The pH of the water in seven buckets were maintained from 3-9 throughout the period of experiments by decanting the water daily and refilling with pH adjusted new water. After 24 h, the leachate was collected and filtered through a Whatman filter paper No.1. The parameters such as color, pH, COD, VFA and polyphenol as lignin and tannin were estimated. The negative control used in this study was husk immersed in normal tap water.

### 2.2.2 Effect of aeration on coconut husk retting

In this study, two set ups were made. In the first set up, fresh coconut husk of about 1Kg (11 months old) was procured and immersed in 4 L tap water. The initial pH of water before aeration was noted. Pre weighed husk was subjected to leaching under the influence of air by setting up with a SOBO aquarium air pump with air stones and air tube. In the second set up, fresh coconut husk of about 1Kg was procured and immersed in 4 L deaerated tap water and kept as a control. From the beginning and then every 30 min, leachate was collected and the color, COD (chemical oxygen demand) and polyphenol content were noted. This was continued up to first 240 min in the aerated system and deaerated system. Then after 24 h, only the aerated leachate was collected and filtered through Whatman filter no. 1. Then the parameters of pH, color, COD, VFA (volatile fatty acids) and polyphenols as tannin and lignin were estimated.

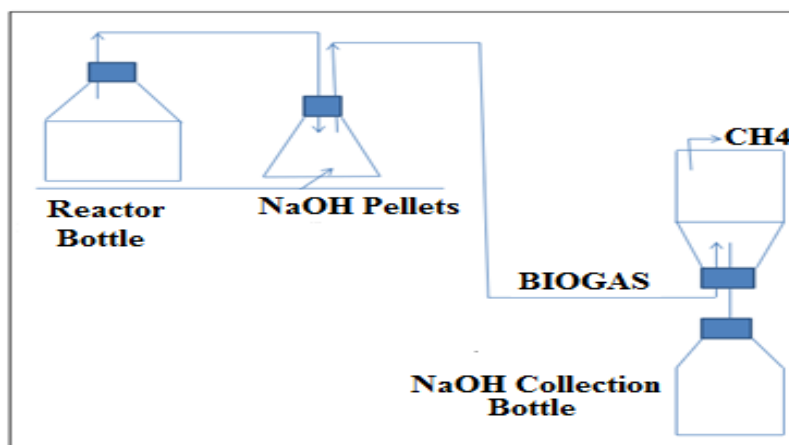
### 2.2.3 Determination of weight loss

Weight difference during leaching was taken as a gross index to explain the magnitude of leaching and was expressed in mg substance soluble on a dry weight basis. Pre weighed fresh coconut husk (11 months old) was procured and immersed in water at different pH. The initial moisture content of the husk was noticed by keeping representative samples of husk at 103°C until a constant weight was attained. Pre weighed husk was subjected to leaching under different pH (3-9). After one week the pre weighed husk samples were taken from each bucket and kept in oven at 103°C until a constant weight (dry weight) was attained. By knowing the initial wet weight and dry weight of the husk, the percentage of weight loss was calculated [9, 10].

### 2.2.4 Testing degradation capability of aerated leachate in anaerobic system

The methanogenic activity test was done in a batch set up based on a displacement system in which carbon dioxide produced is absorbed in NaOH pellets and the methane is collected by the displacement of NaOH solution. The experiment was done in a 500 ml glass bottle. Prior to the actual experiment, a leak test was conducted by filling tap water into 2 bottles. The connections were made using silicon tubing (6 mm OD), and the glass tubes were inserted with rubber corks. The inverted bottle and collection bottle were filled with 3% NaOH solution. All the concentrations were made in the experimental set up and observed for one day to ensure that the apparatus was leak proof (Figure 1).

Initially, the volatile suspended solids (VSS) of the sludge were calculated and an equal amount of biomass was adapted for the husk leachate and control. The initial concentration of substrate was fixed after calculating the COD. The sludge was balanced with nutrient solution, trace elements and substrate for degradation. The final volume of 500 ml was made up by adding deaerated distilled water. The composition of the nutrient solution was  $\text{NH}_4\text{Cl}$  (1.4 g/l),  $\text{KH}_2\text{PO}_4$  (1.25 g/l),  $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$  (0.5 g/l),  $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$  (0.05 g/l),  $\text{NaHCO}_3$  (2 g/l), and Yeast extract (5 g/l). Trace elements added were  $\text{FeCl}_3$  (2 g/l),  $\text{MnCl}_2 \cdot 2\text{H}_2\text{O}$  (0.5 g/l),  $\text{Mg}_2\text{EDTA}$  (0.5 g/l),  $\text{NaSeO}_3$  (0.5 g/l),  $\text{H}_3\text{BO}_3$  (0.1 g/l),  $\text{ZnCl}_2$  (0.05 g/l),  $\text{NH}_4\text{MoO}_4 \cdot 4\text{H}_2\text{O}$  (0.05 g/l),  $\text{Al}_2\text{Cl}_3$  (0.05 g/l),  $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$



**Figure 1.** Schematic diagram of methanogenic activity setup

(0.05 g/l), and  $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$  (0.05 g/l (Merck-India Ltd, Mumbai)). Then, the reactor bottle was connected to a conical flask containing NaOH pellets for the purpose of absorption of moisture and carbon dioxide which was produced during methanogenesis. Biogas production was collected in the inverted conical flask whereas NaOH was accumulated in the NaOH collection bottle. The initial weight of the collection bottle was noted after completing the set up. The weight of the collection bottle was measured daily.

## 2.3 Analytical methods

The Standard Methods (APHA, 1995) was followed during analysis.

### 2.3.1 Determination of color

The change in color intensity was determined using a spectrophotometer (Systronics Digital Bench Top Single Beam UV Visible Spectrophotometer, 195- 1000 nm) at 700 nm.

### 2.3.2 pH Measurements

The pH value of the leachate was measured using a pH meter (Systronics pH system 362). The pH meter was calibrated using pH 4, 7 and 9, respectively.

### 2.3.3 Volatile fatty acids and alkalinity measurement

A simple alkalimeter method was carried out to determine total volatile fatty acids concentration in the anaerobic digester by two stage sequential titration method using volatile standard solution of volatile fatty acids and bicarbonate over a pH 5.5 to 7.65.

### 2.3.4 Estimation of polyphenol

Polyphenol compounds contain aromatic hydroxyl groups that react with Folin's Phenol reagent to form a blue color suitable for estimation of concentration up to at least 9 mg/L. They were estimated spectrophotometrically at a wavelength of 700 nm.

### 2.3.5 COD analysis

The COD tests were carried out according to the open reflux method described in Standard method [11].

### 2.3.6 Total solids test

Total suspended solids and volatile solids were detected by an evaporation method.

### 2.3.7 Determination of dry weight

Coconut husks were oven dried at 103°C to a constant weight and the dry weight (g) was determined.

## 3. Results and Discussion

### 3.1 Effect of pH on coconut husk retting

The leaching property of husk at different pH showed a variation in the amount of leaching with varied pH conditions. It was determined from the level of cumulative COD, polyphenol and VFA as shown in Table 1. Neutral and slightly acidic pH conditions were found to be favorable for higher levels of leaching of organic substrates from husk. Under alkaline pH (pH 9), the leaching was significantly lower. This was a good indication of better leaching process in neutral pH. Weight loss of husk during soaking in water was taken as a gross index of all physicochemical events, and expressed as comparative weight difference from initial moisture content, as shown in Table 2.

It is evident from the results that acidic nature of the soaked liquor caused decrease of pH in the early phase of soaking, and such levels of acidification were not observed in later days. The color of the leachate at different pH and at different time intervals is shown in Figure 2. It is clear from the results that the color of the leachate was higher in the initial days and decreased on the subsequent days of retting in all pH environments. The highest intensity of color was observed at pH7 (0.05) after 24 h, and the lowest intensity of color was at pH3 (0.03) for the same duration. The leachate from all other pH (3-9) showed an intensity of 0.04. The change in pH of all samples that leached out at different pH (3-9) was observed and the results are represented in Figure 3. It is evident from the results that acidic nature of the soaked liquor caused the decrease in pH in the early phase of soaking, and such levels of acidification were not observed in later days. The variation in the VFA profile of leachate for each day is shown in Figure 4, and it is clear that the VFA generation in the system occurred on all days, and the cumulative VFA released (Table 1) was highest at neutral pH (10.75 mEq/L) and lowest at pH 3 (6.20 mEq/L). Daily COD values of the husk-soaked liquor are shown in Figure 5, and it can be seen that the leaching profile of organic materials from husk was high in the initial days and was reduced in later days. The highest cumulative COD value (Table 1) was noted at pH 7 (5736.88 mg/L) and the lowest value was observed at pH 9 (2886.0 mg/L). It is clear that the COD values varied with the pH of the soaking water. The polyphenol concentration released during different days and at different pH conditions is provided in Figure 6. A positive correlation has been reported between extent of retting and the rate of disappearance of polyphenol and pectin [12]. The highest concentration of cumulative polyphenol leached out was at pH 7 (2682.6 mg/l) and lowest concentration was at pH 9 (1685.5 mg/l). This indicates that initial pH of husk soaking liquor has an influence on the release of polyphenols from husk. So, the results showed that the favorable pH for the release of compounds from coconut husk and thereby degradation to extract fiber was neutral. The polyphenol released per kilogram of husk at different pH is shown in Table 3.

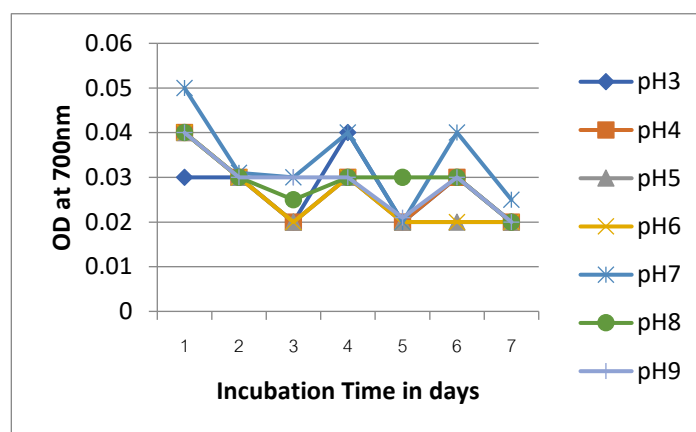
**Table 1.** Showing the variation of characteristics of leachate at different pH conditions

Sl No.	pH treatment	Cumulative VFA (meq/L)	Cumulative COD (mg/ml)	Cumulative polyphenol (mg/L)	Total Polyphenol (mg/L)	Percentage of weight loss
1	3	6.29	4713.8	2313.9	2313.9	4.0
2	4	9.97	5457.0	2542.5	2542.5	5.9
3	5	8.68	4815.6	2456.9	2456.9	4.5
4	6	8.00	4765.3	2242.5	2242.5	6.2
5	7	10.75	5736.9	2682.6	2682.6	6.2
6	8	10.26	4576.3	2068.4	2068.4	4.7
7	9	9.09	2885.3	1685.5	1685.5	3.1
Mean		9.00571429	4707.17143	2284.61429	2284.61429	4.94285714
SD		1.52999844	910.504098	332.389793	332.389793	1.19841165

Standard value for COD is 250 mg/L

**Table 2.** Weight loss of husk at different pH

Sl.No.	pH treatment	Initial Weight of husk (g)	Final weight of husk (g)	Moisture content (g)	%Weight loss
1.	3	88.2	24.6	72.1	4
2.	4	59.2	17.6	70.2	5.9
3.	5	64.1	18.2	71.6	4.5
4.	6	56.2	16.9	69.9	6.2
5.	7	51.2	15.4	69.9	6.2
6.	8	65.1	18.6	71.4	4.7
7.	9	72.3	19.5	73.02	3.08

**Figure 2.** Variation of color with time

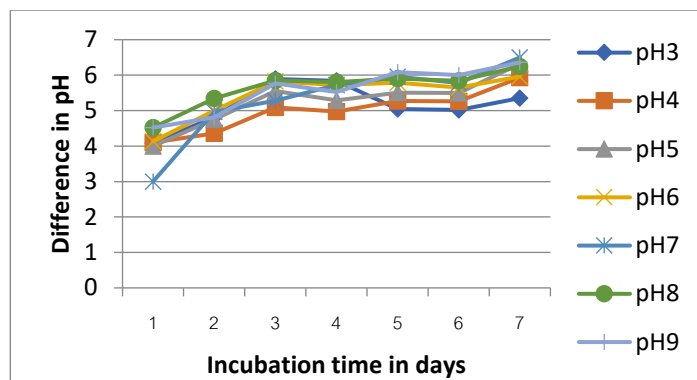


Figure 3. Variation of pH with time

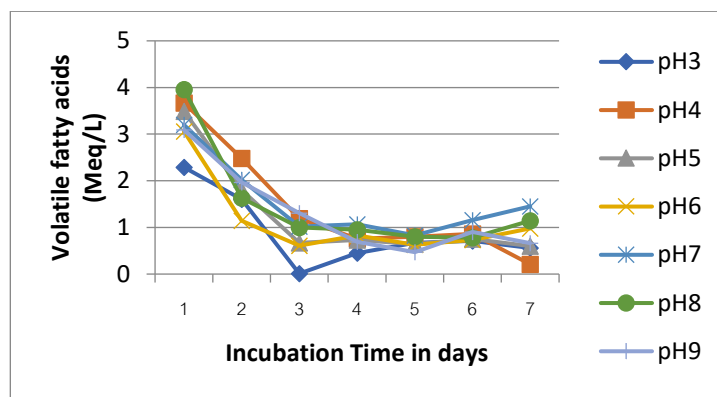


Figure 4. Variation of VFA with time

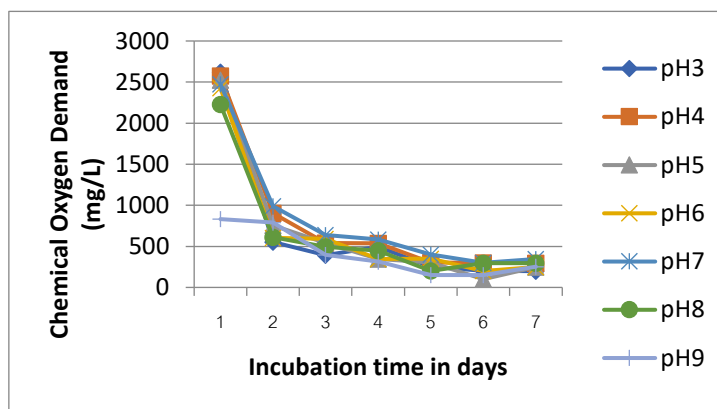
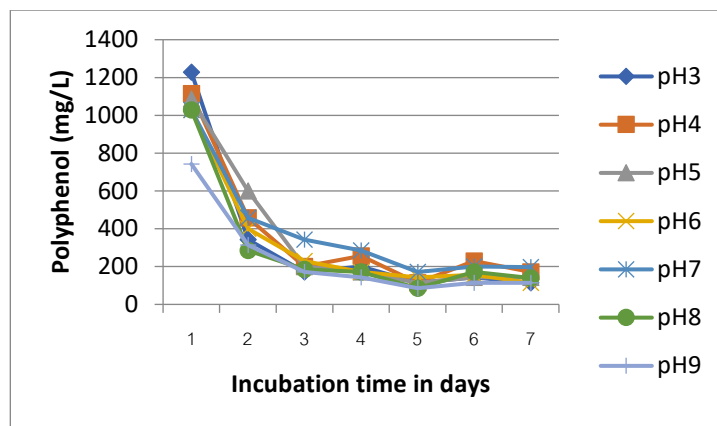


Figure 5. Variation of COD profile with time



**Figure 6.** Variation of polyphenol in different day's interval

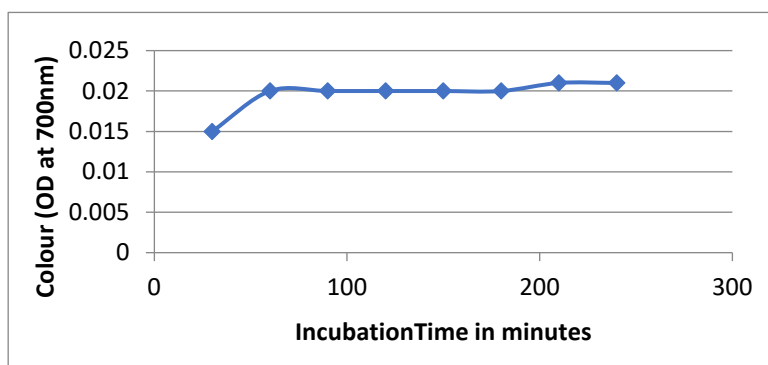
**Table 3.** Total polyphenol leached out per kilogram of husk

Sl.No.	pH	Total polyphenol(g/Kg) of husk
1	3	10.56
2	4	12.06
3	5	11.41
4	6	12.20
5	7	13.26
6	8	10.66
7	9	08.93
Mean		11.29714286
SD		1.403290351

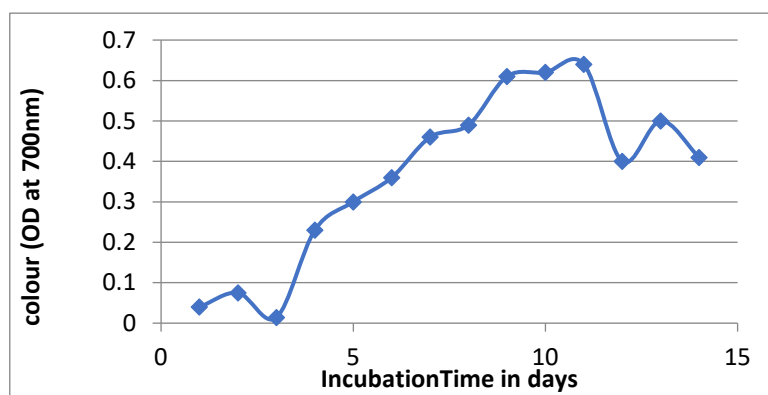
### 3.2 Effect of aeration on coconut husk retting

Color estimation of soaked liquor collected from coconut husk subjected to aeration was done up to 240 min initially and then up to 14 days. The color change was prominent in the initial 30 min and then slowed during the rest of the period. After a week, the color of the leachate was highly intense, and is shown in Figure 7 and Figure 8. Due to aeration, the auto oxidation of tannin compounds takes place. That is why the color of leachate changed to red, and the low molecular weight of tannin changed to high. The auto oxidation of tannin compounds causes slightly red colored substance according to the early reports [13]. Cain in 1980 [14] observed that the auto oxidation of tannin compounds doubled the conversion efficiency bark extract COD to methane (19-40%) and the initial auto oxidation caused 98% enhancement of the biodegradable fraction in tannin rich effluent. The soaked liquor collected from husk under aeration showed a reduction of pH from 6.7 to 5.6 within 14 days, as shown in Figure 9. During this period a rise and fall in the pH between 6.2 and 7.2 was observed, and at the end of the experimental period a sudden fall in pH was noted. The microscopic examination of ret liquor on those days showed an abundant increase in microorganisms, which probably degraded the organic substances in the leachate and produced volatile fatty acids. Therefore, the study revealed that the aeration of soaked liquor could support

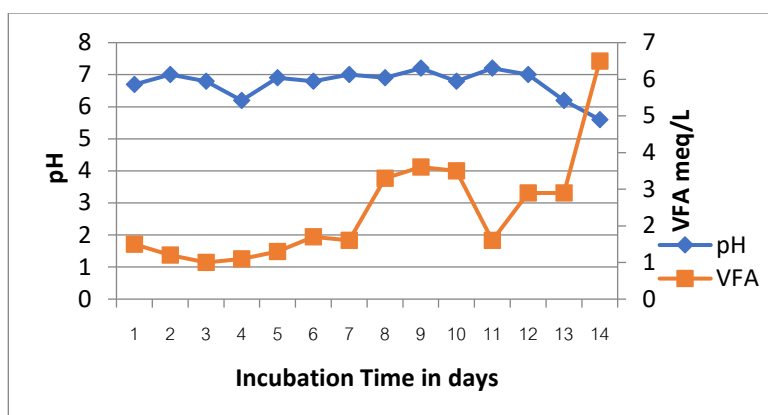




**Figure 7.** Variation of color within first 240 min of aerated leachate



**Figure 8.** Variation of color within 2 weeks of retting

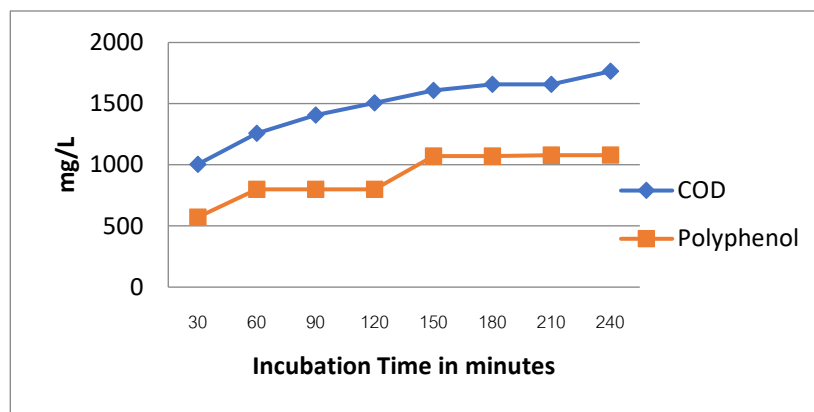


**Figure 9.** Variation of pH and VFA within 2weeks of retting

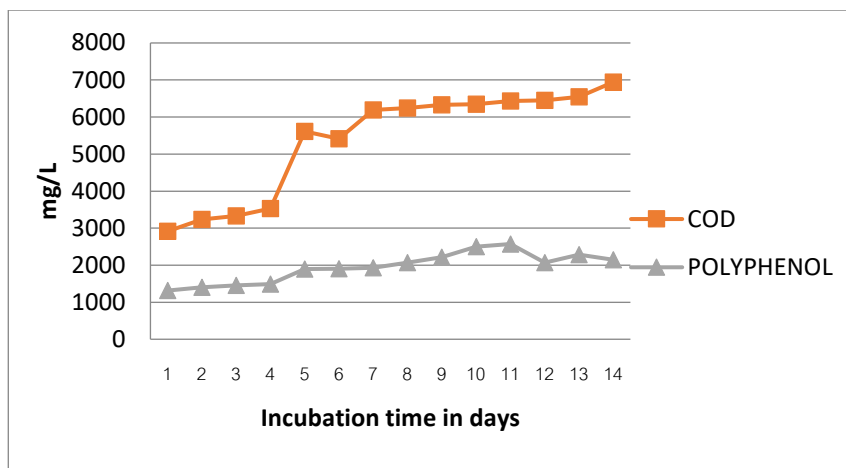
the growth of bacteria that can enhance the retting process [15]. The results regarding volatile fatty acid analysis shown in Figure 9 points out that the VFA level underwent a slow and steady increase throughout the first week of retting. In the second week, a fluctuation of VFA was observed. On the 14<sup>th</sup> day, the VFA was observed to be 6.5 mg/L. Increase in VFA is due to the release of high amounts of acidic substances via the action of hydrolytic microorganisms. The standard pH of water is 7.5 and VFA is considered to be very low in milli equivalents per liter. A high VFA concentration provides conditions which are known to be thermodynamically unfavorable for further degradation of phenolic compounds [16]. The COD analysis also gives a rough idea about the organic material leached out from the coconut husk during soaking. The total COD leached out within 240 min of aerated leachate as shown in Figure 10, and the COD profile of aerated leachate within 2 weeks is shown in Figure 11. The results suggest that under the aerated conditions during the first 240 min, a constant increase of COD values happened. Moreover, the aerated leachate COD increased day by day. The standard or acceptable COD value is about 250 mg/L. In the first 240 min, the aerated leachate had half the COD of non-aerated leachate (Table 4). The polyphenol concentration of the leachate increased daily. The results are shown in Figure 10 and Figure 11. In the first few min, the polyphenol concentration was found to be higher in the aerated condition than in the non-aerated system. So from this result, we came to the conclusion that the microbial action not yet actively started in this time, and that more time is needed for the flourishing that leads to degradation of polyphenol.

**Table 4.** Estimated polyphenol during the first 240 min of soaking of husk under aerated and non-aerated conditions

Sl. no.	Time in minutes	COD of aerated leachate (mg/L)	COD of non-aerated leachate (mg/L)
1	30	1004.0	1612.8
2	60	1256.0	2116.8
3	90	1406.0	2116.8
4	120	1507.2	3024.0
5	150	1607.2	3162.0
6	180	1657.9	3326.4
7	210	1657.9	3402.0
8	240	1764.0	3427.2



**Figure 10.** Variation of COD and polyphenol within 240 min in aerated leachate



**Figure 11.** Variation of COD and polyphenol within 2 weeks of retting

### 3.3. Degradation capability of aerated leachate in anaerobic system

The degradation capability of aerated leachate was studied in a batch system. For 15 days, aerated leachate was added as substrate for anaerobic sludge. The initial characteristics of the aerated leachate are given in Table 5.

**Table 5.** Initial characteristics of the leachate

Coconut husk leachate	Characteristics
pH	7.12
Polyphenol	2462.8 mg/L
COD	6767.3 mg/L
Color at 700nm	0.60

The biomass index of the sludge was determined as volatile suspended solids (VSS) and was 25 mg/L. An equal amount of sludge was added to control and leachate. The entire solution appeared red in color because of the color of the leachate. The initial pH of the control and the leachate were noted as 7.33 and 7.12 and no gas production was observed during the 2 weeks of study. The experiment reveals that the anaerobic sludge was either not capable of utilizing the leachate, or the sludge was not active due to some inhibitory effects of compounds in the leachate. It was also observed that organic and inorganic matters in the wastewater depend on the utilization of biomass activity [17, 18]. Evaluations of sludge extent of digestion can be made from its volatile solids content and its ability to dewater [19]. Studies also reported that inoculum pretreatment of sludge can regulate the volatile fatty acid accumulation [20]. Anaerobic biodegradation is a complex process involving many microbial steps that are conducted by different groups of bacteria, and the toxicity that occurs during anaerobic treatments typically occurs as a result of the incubation of methanogenic/acetogenic bacteria.

## 4. Conclusions

In conclusion, the results obtained can be considered to be important highlights for the selection of favorable pH and aeration conditions for better coconut husk leaching process. Coir retting causes major concerns for the environmental as well as water pollution. The results of the study showed that among the different pH adjustment to (3-9), a neutral pH gave the best leaching and maximum weight loss. This result indicates that under the conditions at which the leachate had the best COD removal in reactor, that was in the anaerobic system with maintenance of aerated conditions, the color of the leachate changed to red in a few minutes, a result which was not seen in the deaerated conditions. The COD values under deaerated conditions were higher when compared to aerated conditions. The steep rises in color and VFA that were observed in the aerated liquor might be due to the high concentration of organic materials in the husk. The biomethanation test on the aerated sample showed no methane production. Like all biological process, anaerobic digestion process is highly reactive to toxicants than aerobic digestion. Similarly, a variety of compounds can inhibit the digestion and cause failure during the process when compared to aerobic process. Moreover, the methanogens are slow growing organisms and the consequence of a toxic incident is far greater in anaerobic system than in an aerobic treatment system. High molecular weight polymeric phenolic components like tannins formed by the auto oxidation of phenolic compounds due to aeration. They are less biodegradable than monomeric phenols. So, further studies are needed to find out how to reduce the influence of toxic components during the anaerobic degradation process. The influence of aeration in the proliferation of micro flora was also a marked feature in this study.

## 5. Acknowledgements

The authors are thankful to Dr.V.B Manilal, Sr. Principal Scientist, Environmental Technology, CSIR-NIIST, Thiruvananthapuram for his help during the course of this work.

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