

Biogas Production through the Anaerobic Digestion of Date Palm Tree Wastes - Process Optimization

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A process for the production of biogas through the anaerobic digestion (AD) of date palm tree waste (DPTW) was developed. The effects of different substrate pretreatments and operating conditions on the yield of biogas and on the chemical composition of DPTW before and after AD were studied. The best results were obtained using alkali pretreatment, with a particle size of 2 to 5 mm, a C:N ratio of 30:1, a digestion temperature of 40 °C, an initial pH of 7.0, and a volatile solids concentration of approximately 10%. The production of flammable biogas containing up to 50% methane started after about one week of operation and continued for approximately 11 weeks. The highest average biogas yield obtained was 342.2 L gas/kg of volatile solids fed to the digester. The highest maximum and average volumetric biogas production rates obtained were 674.5 and 404.4 L/m³ of digester volume per day, respectively. After digestion, there was up to a 58% reduction in the organic matter content of the substrate. Reductions in the contents of cellulose, hemicellulose, and soluble organic compounds were 68.7, 73.4, and 71.9%, respectively, while the ash and lignin contents remained mostly constant. The remaining sludge contained nutrient minerals and some organic matter which qualifies it as a potential soil fertilizer for crop production.

Keywords: Date palm tree waste; Alkali treatment; Soaking; Anaerobic digestion; Biogas; Organic matter reduction

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INTRODUCTION

Anaerobic digestion (AD) has proven to be an efficient method for organic waste treatment. In addition to the production of biogas as green energy, there are additional benefits of this process. These include the production of organic manure, a reduction in toxic and pathogenic effects, the reduction of odor and greenhouse gas emissions, and a reduction in the germination of weeds (Yiridoe *et al.* 2009). In the process of AD, microorganisms convert solid organic matter into methane and carbon dioxide gases. The process is complex and involves a range of microbial associations living in symbiotic relationships.

AD can be divided into 4 phases, within each of which one group of bacteria is active: (a) polymer hydrolysis, *i.e.*, hydrolysis of lignocelluloses into simple sugars;

(b) acidogenesis, *i.e.*, conversion of sugars into acetate and non-acetate fatty acids; (c) acetogenesis, *i.e.*, conversion of non-acetate fatty acids into acetate; and (d) methanogenesis, *i.e.*, conversion of acetate into methane (IWA, 2002). The polymer hydrolysis is considered the first rate-limiting step in AD processes (Appels *et al.* 2008; Yadvika *et al.* 2004), hence pretreatments of lignocellulosic materials are essential to enhance the digestion efficiency of microbes.

Saudi Arabia is one of the largest growers of date palm trees (*Phoenix dactylifera* L.) in the world. It has approximately 24 million date palm trees, with an annual production of about one million tons of fruit (FAO 2012). Date fruits are historical staples in Middle Eastern countries in addition to their cultural and religious significance. There is continuous attention from public and private sectors towards maintaining high levels of date palm production. Approximately one million tons of waste, with high organic matter content, is generated annually from these date palms. This opens the opportunity to seek disposal solutions. A new trend in Saudi Arabia is the use of anaerobic digestion to dispose of organic wastes with the side benefits of producing biogas as a green energy source and sludge as a soil fertilizer. In a study conducted by Al-Juhaimi *et al.* (2014), it was reported that the sludge produced after AD of date palm wastes can be utilized as a fertilizer for crop production.

The objective of this study was to develop a digestion process for the efficient utilization of DPTW for biogas production. The process was optimized, and biogas productivities and yields were measured. In addition, the effect of digestion on the chemical composition of the feedstock was determined.

EXPERIMENTAL

Materials

The feedstock used in this study was date palm tree waste (DPTW) obtained from an establishment for the disposal of dead palm trees in Hofuf City, Saudi Arabia. This feedstock was a product of crushing the whole tree into thin strips with the help of a huge machine.

Digester System

Anaerobic digestion was carried out in a system designed by the authors. It consisted of a digester made of a conical-shaped stainless steel vessel with a working volume of 5 L. The digester was placed in a water bath to control temperature and connected to two Erlenmeyer flasks via rubber tubes for gas collection. The system was properly sealed to ensure anaerobic conditions and to prevent gas leakage. The digester was shaken manually 4 to 6 times a day to homogenize the contents.

Starter Culture

The starter culture was originally obtained from cow dung, which naturally contains methane-producing bacteria. The cow dung was further digested by natural microflora at 40 °C. The starter obtained from this process was used to inoculate first runs of date palm substrates. Subsequent runs were inoculated by back-slopping from the previously digested date palm substrates.

Pretreatments of Substrate

The substrate particle size was reduced to 2-5 mm after milling and passing through a sieve with a pore size of 5 mm. The milled substrate was then pre-treated with alkali (NaOH) at different concentrations (0.75, 1.0, and 1.25%) and soaked for 2, 4, and 7 days, after which the pH was adjusted before digestion to 7.0 using HCl. The C:N ratio was adjusted to approximately 30:1 (Barnett 1978; Bardiya and Gaur 1997) by the addition of ammonium phosphate (containing 21.5% nitrogen) after estimating the C:N ratio of date palm tree waste. The chemical analysis showed that the waste from date palm trees contained 80% carbohydrate (approximately 40% carbon) and 1.5% protein (approximately 16% nitrogen).

Anaerobic Digestion

The milled substrate was carefully suspended in tap water (approximately 10% dry matter) and divided into three containers, followed by the addition of alkali and soaking for the specified time. The pH value was adjusted to 7.0 (Appels *et al.* 2008), the C:N ratio was adjusted to approximately 30:1, and then the substrate was transferred to the digester. A 10% starter was added, followed by digestion at 40 °C. The digester was shaken manually 4 to 6 times a day for about one minute to mix contents. The amount of biogas produced and its methane content were determined.

Chemical Analysis

Cellulose, hemicellulose, lignin, and ash were determined according to Van Soest *et al.* (1991). The organic matter content (volatile solids) were determined using an AOAC method (AOAC, 2000). The biogas was analyzed using a biogas analyzer (Geotechnical Instruments, UK) by measuring the methane concentration using infra-red absorption. The instrument was calibrated using certified methane mixtures (5 to 60% methane content) provided by the manufacturer. A sample tube was connected to the inlet port of the instrument and the results were collected.

Statistical Analysis

All experiments and analytical measurements were carried out in triplicate. The MINITAB statistical package, release 13.30 was used for descriptive statistics, while SPSS package, release 15.0 was used for Duncan Multiple Range Test. A significance level of $P < 0.05$ was used.

RESULTS AND DISCUSSION

Anaerobic Digestion of Feed Stocks after Alkali Treatments

The optimum digestion temperature (40 °C), substrate particle size (2 to 5 mm) and volatile solids concentration in the digester (10%) were determined in preliminary experiments (results not shown). The optimum initial pH (7) and C:N ratio (30:1) were taken from the literature (Appels *et al.* 2008, Barnett 1978; Bardiya and Gaur 1997). The results of the anaerobic digestion (AD) experiments with milled date palm tree waste (DPTW) substrates containing 327.6 g VS and treated at NaOH concentrations of 0.75%, 1.0%, and 1.25% (keeping all other AD parameters constant) are shown in Figs. 1 and 2. The production of flammable gas, *i.e.*, gas containing up to 50% methane, started after about a week of AD and continued until week 11 (Fig. 1). Production from the 0.75%

NaOH treatment was the highest from weeks 1 to 3, then it gradually dropped until the end of the experiment. Production from the 1.0% NaOH treatment increased steadily until week 5, then it dropped sharply; whereas production from the 1.25% NaOH-treated DPTW was relatively steady until week 8. The average total amount of gas produced in the 11 weeks of operation was approximately 112, 107, and 108 L from 0.75, 1.0, and 1.25% NaOH-treated substrates, respectively (Fig. 1), all of which contained 50 to 60% methane. There were no significant differences ($P>0.05$) between these amounts. Nearly 70% of the gas was produced in the first 6 weeks in case of the 0.75 and 1.0% NaOH treatments, while in case of the 1.25% NaOH treatment, this amount was produced in approximately 7 weeks of operation (calculated from Fig. 1). It can therefore be inferred that processes with 0.75 and 1.0% NaOH treatments continue with optimal production for 6 weeks, while production from the 1.25% NaOH treatment can extend for approximately 7 weeks with fairly constant biogas productivity. Substrates that were not treated with NaOH produced negligible amounts of biogas. Similarly some other combinations of NaOH treatment (0.25, 0.5% and 1.5%) produced less than 50% of the amounts produced using 0.75% to 1.25% NaOH treatments (results not shown). This indicates that concentrations of NaOH that are too low or too high in DPTW pre-treatment for biogas production may not be useful. Alkali pretreatment of lignocellulosic materials improves their biodegradability by opening their compact structure (Taherzadeh and Karimi 2008). Dar and Tandon (1987) observed an improvement of 31 to 42% in microbial digestibility and an almost twofold increase in biogas production when alkali-treated (1% NaOH for 7 days) plant residues were used as a supplement to cattle dung. Alkali treatments, however, are not without problems. In continuous reactors fed with alkali-treated feedstock (the initial concentration of sodium was 0.21 Mol/L), acetate and glucose degradation rates have been observed to fall to 5% and 50%, respectively, due to toxic compounds formed after this treatment (Mouneimne *et al.* 2003).

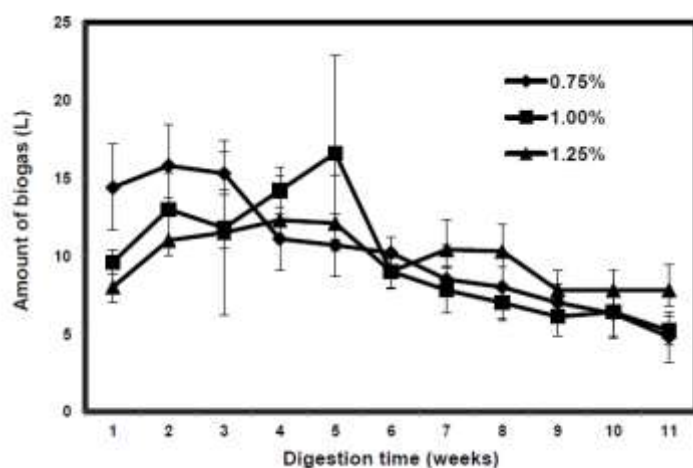


Fig. 1. Biogas produced from date palm tree wastes treated with 0.75%, 1.0%, and 1.25% NaOH and soaked for 7 days (327.6 g volatile solids in 3.6 L volume). The results shown are the means from triplicate runs and are presented with standard error.

The process developed in this study showed biogas productivity that was quite stable over up to 4 consecutive runs (approximately 9 months) using back-slopping for inoculation, indicating the stability of the microbial association involved in the digestion.

However, in two out of approximately 33 runs some disturbance was observed in the process stability; this was shown as a gradual drop in pH from the initial value of 7 down to 4.5-5.0 accompanied by a decrease in biogas productivity until production stopped completely. When the pH was raised to 7, biogas production commenced but decreased again and eventually stopped as a result of another pH drop. This situation indicates an acid accumulation, most probably due to the absence or inhibition of the acetogenic group. Acetogenic bacteria consume the non-acetate fatty acids (produced by acidogens) converting them into acetate, which is then further converted by methanogens into methane (IWA, 2002). Because the non-acetate fatty acids are not directly metabolizable by methanogenic bacteria, they will accumulate in the medium in the absence of acetogenic activity. Hence the pH of the system will decrease and consequently inhibit methane formation by methanogens which are very sensitive to low pH (Garcia *et al.* 2000).

The 0.75% NaOH treatment resulted in the highest biogas yield with an average of approximately 342 L gas/ kg VS fed to the digester. The other two treatments gave yields close to each other, with averages of approximately 326 and 320 L gas/kg VS for the 1.00% and 1.25% treatments, respectively (Fig. 2). The yields of biogas from these three alkali pre-treatments were not substantially different. There were also no significant differences between these three treatments in terms of maximum and average volumetric productivities. Generally, the volumetric productivities remained relatively high from week 1 to week 8 for these treatments and then began to decline.

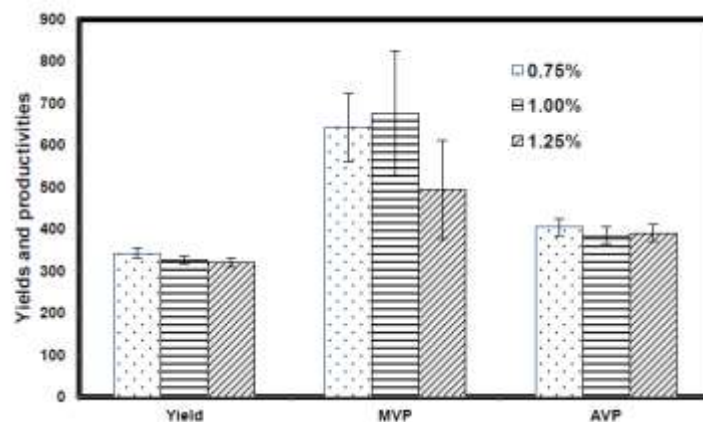


Fig. 2. Yields (L gas/kg VS) and the maximum (MVP) and average (AVP) volumetric productivities (L /m³ digester volume in a day) of biogas produced from date palm tree waste after an alkali treatment and soaking for 7 days (327.6 g volatile solids in 3.6 L volume). The results shown are the means from triplicate runs and are presented with standard error.

The biogas yield from volatile solids and the volumetric productivity (maximum and average) of the system are important parameters. The amount of yield is an indication of the suitability of the substrate for biogas production and is also an indication of the efficiency of the microbes involved in the digestion process. Higher yields mean that less substrate is needed to produce a certain amount of biogas. On the other hand, the volumetric productivity is a measure of the efficiency of the digester system. Higher productivity means that smaller digester volumes are needed for the production of certain amounts of biogas. The maximum productivity indicates the peak of production, while the average productivity indicates the average amount of daily production over a certain

period of time. Both the yield and productivity were affected by the digestion conditions of temperature, pH, and VS concentration, and so on. Our results compare well to results obtained by other researchers. Gupta *et al.* (2013) obtained a yield of 442 L biogas/kg total solids from a substrate made of 50% detoxified mahua seed cake. Monlau *et al.* (2013) obtained yields of 302 L methane/kg VS after acid pretreatment of sunflower oil cake at 170 °C. Hagelqvist (2013) obtained a methane yield of approximately 85 mL/g VS (*i.e.*, approximately 170 L biogas/kg VS) from municipal sewage sludge and approximately 55 mL/g VS from sludge from a pulp and paper mill. Lin *et al.* (2009) obtained the highest biogas yields from anaerobic digestion of pulp and paper sludge pretreated with 1.2% NaOH. Alkali pretreatment was also described by many other researchers as being promising for the improvement of anaerobic digestion of newspaper, corn stalk, hardwoods, softwood, and paper tubes (Fox *et al.* 2003; Teghammar *et al.* 2009; Mirahmadi *et al.* 2010; Salehian *et al.* 2013).

Effects of Alkali Treatment on the Chemical Composition of Substrate

The date palm substrate used in this study contained 76.2% lignocelluloses with 14.3% lignin (Table 1). Before the alkali treatment the particle size of the substrate was reduced to 2-5 mm to improve accessibility to the treatment. The alkali destroys the lignin shell protecting the cellulose and hemicellulose, decreases the crystallinity of cellulose, and increases porosity (Dakar 2014). The NaOH treatment in this work caused a slight decrease in the cellulose and hemicellulose contents, an increase in the soluble solids content (sugars, amino acids *etc.*), and no change in the contents of ash and lignin (Table 1). A limited amount of hydrolysis of cellulose and hemicellulose occurred, indicating that they were exposed to the action of alkali, while lignin seemed not to be broken down into simple components by this treatment. An increasing alkali concentration increased the cellulose hydrolysis, and its content decreased from 42.4% in the untreated sample to 38.9, 37.2, and 33.4% in the samples treated with 0.75%, 1.0%, and 1.25% NaOH, respectively. The increase in the amount of soluble solids is regarded as a result of cellulose and hemicellulose hydrolysis. Unlike the action on cellulose, increasing the NaOH concentration from 0.75% to 1.25% did not show an increase in the amount of hydrolysis of hemicellulose (Table 1).

Table 1. Effect of Treatment with NaOH (7 days soaking) on the Chemical Composition of Date Palm Tree Fibers

Component (% dry basis)	Treatment (% NaOH added)			
	0.0	0.75	1.0	1.25
Cellulose	42.4	38.9	37.2	33.4
Hemicellulose	19.5	12.3	12.0	12.7
Lignin	14.3	15.1	14.9	14.3
Soluble solids	16.1	27.5	29.5	32.7
Ash	5.4	4.7	5.6	4.5

Effects of the Duration of Soaking After NaOH Addition on the Anaerobic Digestion of DPTW

The soaking of DPTW after the addition of NaOH for different time durations before digestion resulted in variable effects on biogas yield and on the organic matter reduction (OMR). In the case of the 0.75% NaOH-treated DPTW, the biogas yield

increased significantly with increasing soaking time from 269 (2 days soaking) to 305 (4 days soaking) and 342 L /kg VS (7 days soaking). Also the amount of OM reduction increased significantly ($P<0.05$) with increasing soaking time from 45.3% (2 days soaking) to 50.8% (4 days soaking) and 58.2% (7 days soaking), as shown in Table 2. The biogas yields increased with increasing soaking time in the case of 1.0% NaOH-treated DPTW, but the difference became significant only after the 7 days soaking time. Although there were increases in OMR for the 1.0% treatment and in the yields and OMR for the 1.25% treatment with increasing soaking time, the differences were not significant (Table 2). It appears that the relatively mild 0.75% NaOH treatment needed more time to bring about the desired effect, while the other stronger treatments needed less time. The reduced yields from the 1.0% and 1.25% treatments compared to the 0.75% treatment indicate that some form of inhibition to the microbial population started to appear as the NaOH concentration increased.

Table 2. Effect of Soaking Duration after NaOH Treatment of Date Palm Tree Waste on Biogas Yield and Organic Matter Reduction (OMR)

Soaking (Days)	NaOH (%)					
	0.75		1.0		1.25	
	Biogas yield (L gas/kg VS)	OMR (%)	Biogas yield (L gas/kg VS)	OMR (%)	Biogas yield (L gas/kg VS)	OMR (%)
2	269±9.9 ^c	45.3±2.1 ^c	299±13.0 ^b	47.9±2.2 ^a	301±18.1 ^a	52.0±6.8 ^a
4	305±13.4 ^b	50.8±2.0 ^b	311±9.9 ^{ab}	48.8±1.6 ^a	314±12.5 ^a	52.8±5.8 ^a
7	342±11.3 ^a	58.2±2.0 ^a	326±9.2 ^a	50.5±1.4 ^a	320±10.1 ^a	54.9±6.4 ^a

VS = volatile solids. Means (± standard deviation) within columns having same superscript letters are not significantly different ($P<0.05$)

Changes in Organic Matter Content and Chemical Composition of Date Palm Tree Substrates after Anaerobic Digestion

The anaerobic digestion of DPTW resulted in an organic matter (OM) reduction of 58.2%, 50.5%, and 54.9% after NaOH treatments of 0.75%, 1.0%, and 1.25%, respectively and soaking for 7 days before digestion (Table 3). Compared to the other two treatments, the 1.0% NaOH treatment resulted in significantly ($P<0.05$) lower OM reduction. The 0.75% treatment was superior to the other treatments in both biogas yield and OMR, and the 1.0% treatment was superior to the 1.25% in biogas yield but not in OMR (Tables 2 and 3). This can be explained as follows: the bacteria in the 1.25% treatment consumed more OM, hence the increased OMR, but may have converted the greater part of it into metabolic products other than methane, hence reducing the biogas yield. The individual components of the OM of DPTW were affected at different levels as a result of anaerobic digestion (Tables 4 and 5). The relative contents of cellulose, hemicellulose, and soluble solids decreased, while the content of lignin increased (Table 4). This shows that cellulose, hemicellulose, and soluble solids were consumed by the microorganisms, while lignin was not. The concentrations of these components in the substrate before and after AD were calculated for the 3.6 L digestion volume used in these experiments. Hence the total organic matter in the 3.6 L AD volume for the three

treatments was 327.6 g before digestion (Table 5, calculated from the 9.1% content of Table 3). The total amount of organic matter after digestion was 136.8 g, 162.0 g, and 147.6 g for the 0.75%, 1.0%, and 1.25% NaOH treatments, respectively (Table 5, calculated from the 3.8, 4.5, and 4.1% concentrations of Table 3). From the composition of the organic matter in the substrate before and after AD presented in Table 4, the amounts of these components in the 3.6 L digestion volume before and after AD were calculated (Table 5), and from that the amount of reduction in each of them was determined.

Table 3. Effect of Digestion on the Organic Matter (OM) Content of Substrate of Date Palm Tree Waste

NaOH	OM before digestion (% dry matter)	OM after digestion (% dry matter)	OM reduction (%)
0.75%	9.1	3.8±0.18 ^c	58.2±1.99 ^a
1.0%	9.1	4.5±0.13 ^a	50.5±1.41 ^b
1.25%	9.1	4.1±0.14 ^b	54.9±1.56 ^a

Means within columns that have the same letter are not significantly different ($P<0.05$)

Table 4. Effect of Digestion on the Chemical Composition of Date Palm Tree Substrate

Component (% dry matter)	Treatment (NaOH %)					
	0.75		1.0		1.25	
	bAD	aAD	bAD	aAD	bAD	aAD
Cellulose	38.9	29.2	37.2	26.8	33.4	26.0
Hemicellulose	12.3	7.8	12.0	8.7	12.7	8.5
Lignin	15.1	35.0	14.9	31.6	14.3	33.4
Solubles	27.5	18.5	29.5	24.6	32.7	23.8

bAD = before anaerobic digestion, aAD = after anaerobic digestion

The highest reduction in all of the components was encountered in the 0.75% NaOH treatment, followed by the 1.25% treatment. The reduction in cellulose content ranged between 68.7% and 64.4%, in hemicellulose between 73.4% and 64.1%, and in the solubles between 71.9% and 58.7%. The lignin content was not affected, and the apparent increase shown in Table 5 may be a result of analytical errors. Gupta *et al.* (2013) achieved a 33.2% and 34.1% reduction in total solids and volatile solids, respectively, from a substrate made of 50% detoxified mahua seed cake. The cellulose and hemicellulose contents of the digested slurry were reduced by 34.5% and 29.8%, respectively, while the content of mineral nutrients (N, P, K) was increased. Hagelqvist (2013) obtained approximately 32% and 50% degradation of volatile solids of sludge from a pulp and paper mill and from municipal sewage sludge, respectively. The sludge of AD of DPTW in this study was successfully used as soil bio-fertilizer for alfalfa cultivation, indicating potentials for use in crop production (Al-Juhaimi *et al.* 2014). The current study represents a novel attempt at the utilization of DPTW for its potential as a bioresource.

Table 5. Mass Balance of the Organic Components of Date Palm Tree Substrate Subjected to Digestion

Component	Content before digestion (g)	Content after digestion (g)	Change (%)
0.75% NaOH treatment			
Total organic matter	327.6	136.8	- 58.2
Cellulose	127.4	39.9	- 68.7
Hemicellulose	40.3	10.7	- 73.4
Lignin	49.5	47.9	- 3.2
Total solubles	90.1	25.3	- 71.9
1.0% NaOH treatment			
Total organic matter	327.6	162.0	- 50.5
Cellulose	121.9	43.4	- 64.4
Hemicellulose	39.3	14.1	- 64.1
Lignin	48.8	51.2	+ 4.9
Total solubles	96.6	39.9	- 58.7
1.25% NaOH treatment			
Total organic matter	327.6	147.6	- 54.9
Cellulose	109.4	38.4	- 64.9
Hemicellulose	41.6	12.5	- 70.0
Lignin	46.8	49.3	+ 5.3
Total solubles	107.1	35.1	- 67.2

Calculated from the data in Tables 3 and 4; Substrate 3.6 L, with a 9.1% organic matter content

CONCLUSIONS

1. Pre-treatments using NaOH (0.75%, 1.0%, and 1.25%) and soaking the treated substrate for 2 to 7 days before digestion increased biogas yield by positively altering the chemical composition of DPTW.
2. The maximum yield (342.2 L biogas/kg volatile solids) was obtained when digestion was carried out at 40 °C, with an initial pH approximately 7.0, volatile solid concentration of 10%, particle size of DPTW 2-5 mm, C:N ratio 30:1, and alkali pretreatment.
3. The maximum and average volumetric biogas productivities obtained were 674.5 and 404.4 L/m³ digester volume per day, respectively. There was a significant reduction in the organic matter content of the DPTW after digestion, showing that microbes effectively utilized it in their metabolic activities.

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