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# Effectiveness of Palm Kernel Shells as Packings for Biogas Chemical Scrubbing at Ambient Conditions

**Alutu NC, Duru PN, Maduegbunam HU**
Research and Development Department, National Engineering Design Development Institute, PMB. 5082, Nnewi Anambra State, Nigeria

***Corresponding author**
*Alutu NC*

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**Abstract:** Bio-methane is a flammable renewable energy resource processed an-aerobically from biomass which includes municipal wastes, farm animal wastes, and vegetable/food wastes among others. The raw bio-methane produced contains methane gas and some contaminants like Carbondioxide ($CO_2$), Hydrogen Sulphide ($H_2S$), moisture and other trace compounds in varying quantities depending on the source and type of substrate materials. Many researchers have used synthetic and manufactured packing materials for scrubbing biogas which are expensive and needed regeneration for economic purposes. But the use of palm kernel shell as packing material in a packed bed for scrubbing biogas is an innovation for modern technology which proved to be very suitable for biogas scrubbing.
**Keywords:** bio-methane, palm kernel shells, scrubbing, modern technology, packing materials, contaminants

## INTRODUCTION

Bio-methane is a flammable renewable energy resource processed an-aerobically from biomass which includes municipal wastes, farm animal wastes, and vegetable/food wastes among others. The raw bio-methane produced contains methane gas and some contaminants like Carbondioxide ($CO_2$), Hydrogen Sulphide ($H_2S$), moisture and other trace compounds in varying quantities depending on the source and type of substrate materials.

These impurities cause corrosion of metal parts and reduce the heating value of the raw bio-methane if not purified. Bio-methane has a slightly lower heating value than pure methane since the energy density is proportional to the methane content [1]. Methane is odourless and colourless and it is a powerful greenhouse gas, 25 times more potent than carbon dioxide based on the global warming potential over 100 years [2].

In order to fully exploit the bio-methane potential as vehicle fuel or natural gas substitute, bio-methane itself must be upgraded to increase the calorific value. Bio-methane upgrading, that is, the treatment for $CO_2$ scrubbing, can be performed by several techniques, each one characterised by a different energy demand. High pressure water scrubbing (HPWS, where pressure is around 10 bars) is one of the most common techniques applied in an upgrading bio-methane plant for simultaneous scrubbing of $CO_2$ and $H_2S$ [3]. Considering the high energy cost, high pressure drop, flooding and increased capital cost of regeneration and disposal of spent water associated with High pressure water scrubbing, according to Cozma *et al*, [3] and Vijay *et al.* [1]; a chemical method of purification of bio-methane is to be considered. The scrubbing of bio-methane has some influencing factors like packed bed height, gas flow rates, scrubbing time, solvent concentrations among others.

Some packings especially Ring and Saddle are available in a variety of materials: ceramics, metals, plastics and carbon. The choice of material for packed column will depend on the nature of the fluids and operating temperature [4]. Ceramic packing is suitable for corrosive liquids according to Sinnot [4], but not with strong alkalis. He also stated that plastics packing are attacked by some organic solvents and can only be used up to moderate temperatures. For any material to serve as packing material, Ronald *et al.* [15] asserted that voids between individual units must not create resistance to upward gas flow, the shapes of the packings should not form droplets at the edges which do not allow for sufficient contact between the gas and the water; the packing should have good wetting characteristics for water to uniformly distribute itself within the column for the entire packed depth.

The principal requirements of a tower packing according to Sinnot 2005 are:

- It must be chemically inert to the fluids in the tower.
- It must be strong without excessive weight.
- It must contain adequate passages for both streams without excessive liquid hold up or pressure drop.
- It must provide good contact between liquid and gas.
- It must be reasonable in cost.

Most packing is made of cheap, inert, fairly light materials such as clay, porcelain, or graphite. Thin-walled metal rings of steel or aluminium are sometimes used.

Common packings are:

- Berl Saddle.
- Intalox Saddle.
- Rasching rings.
- Lessing rings.
- Cross-partition rings.
- Single spiral ring.
- Double - Spiral ring.
- Triple - Spiral ring.

All the types of packings listed above are manufactured materials but research has not been done on palm kernel shells as a suitable packing material for biogas scrubbing being an agricultural waste.

Scrubbing with Mono-ethanolamine (MEA 10 to 20% w/w solution in water) and its regeneration was operated at atmospheric pressure in packed column with pall rings by Simone *et al.* [5]. They discovered that the three scrubbing methods gave bio-methane purity of up to 98%. Considering other chemicals as absorbent solvent, Manjula [6] used soda lime which he claimed was a mixture of $Ca(OH)_2$, KOH, NaOH and water in varying proportions and was able to achieve only 97.7% of methane which with only NaOH alone one can scrub more than that. This venture may be termed waste of resources. Shah *et al.* [7] also experimented scrubbing bio-methane with mixture of dry lime and KOH and NaOH mixed with $Ca(OH)_2$. They got percentage scrubbed bio-methane as 95.11% and 94.69% respectively which was barely a little up to natural gas standard.

Mohammed *et al*. [8] asserted that removal efficiencies vary for each pollutant-solvent system and with the type of gas absorber used. They stated that while most absorbers have removal efficiencies higher than 90%, the packed tower absorbers may achieve efficiencies as high as 99.9% for some pollutant-solvent systems. Considering influencing parameters like input gas flow rates, packing height on the scrubbing efficiencies, Mohammed *et al.* [14] discovered that higher gas flow rates led to higher turbulent flow, introducing higher energy to the gas which consequently leads to higher removal efficiency. Without packed bed enough contact time was allowed between the bio-methane and water as Hendry *et al.* [9] discovered that the highest reduction of $CO_2$ was gained in slower biogas volumetric flow rate of 1lit/min and higher water volume.

**Fourier Transform Infrared Spectroscopy (FTIR) analysis**

Are an analytical testing technique used to identify organic and some inorganic materials through the application of infrared radiation (IR). Meyer [10] added that FTIR technique can also be used to obtain an infrared spectrum of absorption, emission, photo conductivity or Raman scattering of a solid, liquid or gas. As the sample absorbs the infrared light, the absorbance of energy at the various wavelengths is measured to determine the material's molecular composition and structure. The patterns of absorption bands at the various wavelengths throughout the infrared region (or the FTIR spectrum) are unique to each material. IR spectroscopy is often used to identify structures because functional groups give rise to characteristic bands both in terms of intensity and position (frequency). The sample's absorbance of the infrared light's energy at various wavelengths is measured to determine the material's molecular composition and structure. Abdul *et al.* [11] in "comparison of various sources of high surface area carbon prepared by different types of activation," used FTIR to identify raw palm kernel shell absorption frequencies and functional groups. Abdul *et al*. [11] noted that the O-H functional group stretching indicates the presence of bonded hydroxide in the raw sample. Meyer [10] informed that structural features of the molecules produce characteristic and reproducible absorptions in the spectrum which detects whether there is backbone to the structure or not and if there is backbone, whether it consists of linear or branched chains. Also it detects whether there is unsaturation and/or aromatic ring in the structure as well as the local environment or location in the structure, the origins of the samples of detected functional groups, its pre-history and the manner in which the sample is handled.

Okoroigwe *et al*. [12] stated that morphology and pore structure are important for expelling volatiles when subjected to thermochemical processes such as torrefaction, pyrolysis and gasification. Crystal shape size of the crystalline solid phase could be identified from micrograph. Micrographs reveal the micro pores through which the kernel exchanged fluids with the surrounding mesocarp. Thus Palm Kernel Shells (PKS) has potential as a sorbent material.

**Voidage (void fraction)**

Is the fraction of the total volume made up of free space between the particles which is filled with fluid. Mohammed, 2011 explained further that fractional void is the fraction of the volume of bed not occupied by solid materials and can be called porosity $\varepsilon$. The term void fraction is normally used to refer to the tiny spaces between packed particles in a container. The actual calculation of voidage is simple: it is the amount of empty space divided by the total volume. Calculation of voidage in regular shaped spherical packings as a function of particle and bed diameter can be determined using the Furnas equation stated below according to Mohammed (2011):

$$\varepsilon = 0.375 + 0.34\,\frac{dp}{Dr} \qquad (1)$$

Where,

dp =particle diameter

Dr = bed diameter

For irregular shapes like palm kernel shells, calculation of the void fraction takes the following formula:

$$\text{Voidage \%} = \frac{\text{volume of the water added}}{\text{volume of the container}} \times 100 \qquad (2)$$

Therefore the volume of the bed occupied by solid materials is 1- $\varepsilon$

The increase in void fraction decreases the pressure drop in a packed bed and so decreases the size of pump to push the liquid through the pipe. This was proved from the Kozeny- Carman equation given below in equation 4 according to Mohammed 2011.

$$\frac{\Delta P}{L} = \frac{150\,\bar{v}o\mu(1-\varepsilon)^2}{\Phi_s^2 D_p^2\,\varepsilon^3} \qquad (3)$$

Where

$\bar{v}\,o$ = superficial velocity or empty tower velocity

$\mu$ = fluid viscosity

$\varepsilon$ = porosity

$\Phi_s$ = sphericity of particle

$D_p$ = particulate diameter

Void fraction or porosity is very important according to Mohammed 2011, in that it

- Determines the bulk density of the material and hence the volume taken up by a given mass.
- Affects the tendency for agglomeration of the particles.
- Influences the resistance which the material offers to the percolation of fluid through it.
- Void fraction influences pressure drop for flow through the phase, electrical resistivity, effective thermal conductivity of the phase and reactive surface area.

Porosity of a static bed depends on the following;

- Particle size and size distribution
- Particle shape
- Surface roughness
- Method of packing and
- Size of container relative to the particle diameter.

**MATERIALS AND METHODS**

Biogas was generated and characterized. Then a scrubber was connected to a bio-digester through inlet air hose. The scrubber was ¾ filled with local palm kernel shells of irregular shapes to serve as the packings. These packings were included to increase the mass transfer area of contact between the inlet gas which entered through the bottom and the scrubbing liquid of aqueous NaOH of 1mole concentration. The scrubbing was done at ambient conditions to solve the problem encountered by Cozma et al, 2014 while studying on high pressure water scrubbing system. Analyses were carried out to determine the suitability of using palm kernel shells as packing material for biogas scrubbing in a packed bed. These were

- Fourier Transform Infrared spectroscopy (FTIR)
- Scanning Electron microscope (SEM) micrographs
- Volume void calculation

**Fourier Transform Infrared spectroscopy (FTIR)**

The palm kernel shells were analysed using FTIR-8400S spectrophotometer to identify the types of chemical bonds (functional groups) contained. The device produced an infrared absorption spectrum that is like a molecular “finger print” called peaks.

The solid palm kernel shells samples were dissolved in a methylene chloride solvent and the solution placed onto the plate to create a cast film. The solution was then analysed in a liquid cell which is a small container made from NaCl (or other Infrared (IR) - transparent material). The FTIR peaks were shown in the figure 1 below while the interpretation was shown in tables 1 and 2 representing the chemical bonds of the palm kernel shells before and after scrubbing processes respectively.

**Scanning Electron microscope (SEM) micrographs of fresh and spent palm kernel shells**

The morphology and surface structure of the palm kernel shells (fresh and spent) were exposed using

Scanning Electron microscope (SEM). The filming was done using JEOL scanning electron microscope model JSM 6400. The solid samples were placed on a brace stub sample holder using double stick carbon tape. The samples were coated with layers of gold approximately 25A thick using blazer sputtering coater. This coating was normally done to organic materials (non-conductive materials) using a sputter coater to make them conductive. The micrographs were recorded with 15 KV mapping and 1000x magnification. The micrograph was shown in the figures 2 and 3 for fresh and spent palm kernel shells representing before and after scrubbing processes respectively.

**Volume void calculation**

For irregular shapes like palm kernel shells, calculation of the void fraction takes the following simple steps:

A container of known volume of 2500millilitres was noted. The container was filled and levelled with the palm kernel packings with constant shaking of the container to ensure there was no more space to be taken by the packings. A calibrated measuring cup was filled with water. The initial amount of water started with was noted. Water was added slowly to the container and constant tapping of the container was done several times to dislodge every trapped air. Water was continuously added until the packing becomes saturated and further addition of water would spill out.

Saturation of the packings indicated that water has filled up any empty spaces and the amount of water added was noted.

Calculation:

$$\text{Voidage } \% = \frac{\text{volume of the water added}}{\text{volume of the container}} \times 100$$

$$\text{Voidage} = \frac{1300}{2500} \times 100$$

$$\therefore \ \% \text{ voidage} = 52\%$$

## RESULTS AND DISCUSSION

### Fourier Transform Infrared Spectroscopy (FTIR) Study of the palm kernel shells

The FTIR analysis was used to examine the surface functional groups of the packing material and to identify those groups responsible for gas absorption. Absorption in the IR region takes place because of rotational and vibrational movements of the molecular groups and chemical band of a molecule by Natali *et al* [13].

**Table−1: Fourier transform infrared spectrum for fresh palm kernel shell**

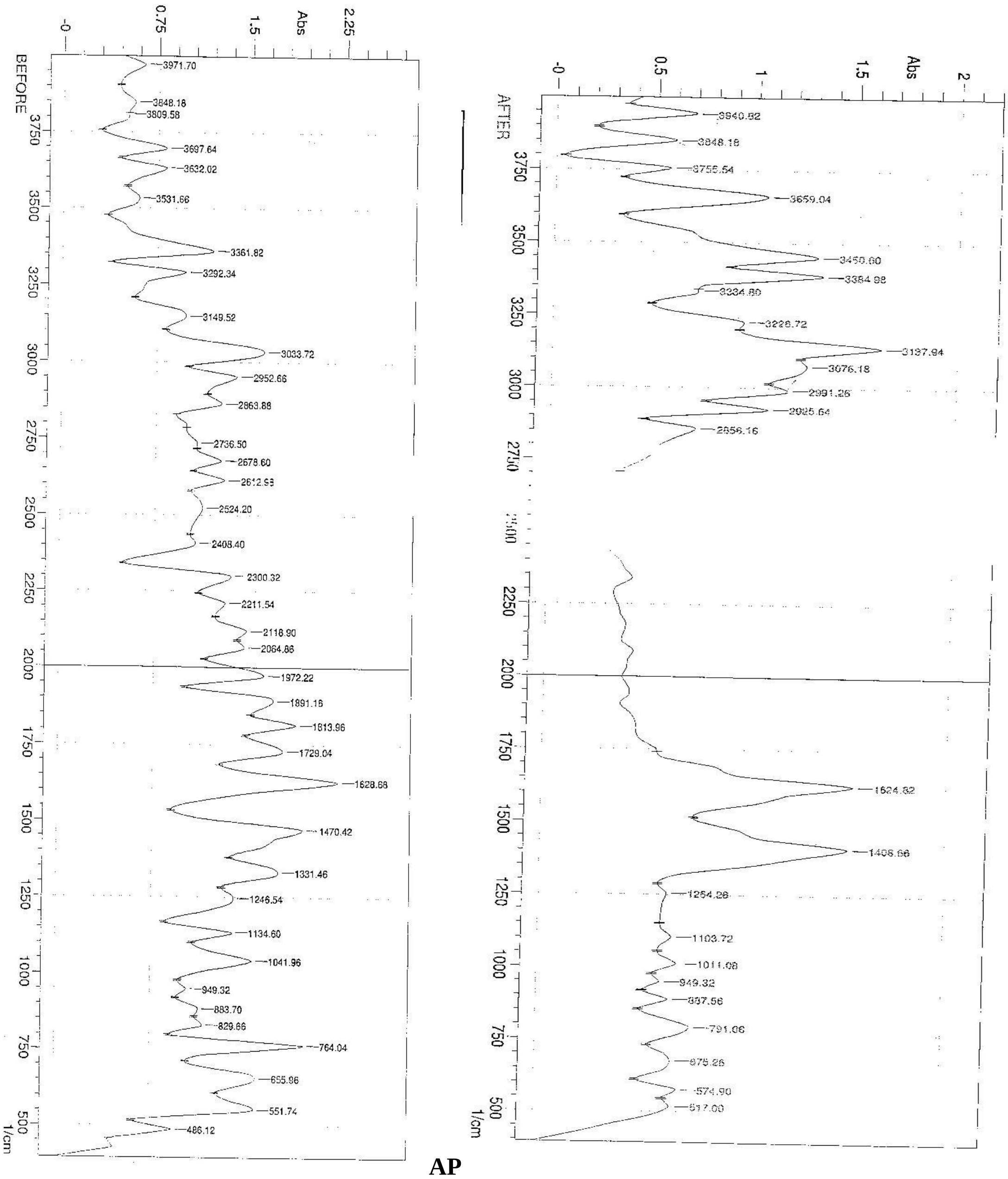
| Wave number $cm^{-1}$ | Bond Source | Functional group |
|---|---|---|
| 3971.70 − 3292.34 | O – H stretch<br>N – H | Amine, Alcohol |
| 3149.52 – 2736.50 | O - H stretch, free | Aldehydes, carboxylic acids |
| 2678.60 – 2408.40 | O – H stretch, very broad | Carboxylic Acids |
| 2300.32 | C = H stretch | Alkenes |
| 2211.54 – 2118.90 | C ≡ H stretch | Alkynes |
| 2064.86 – 1813.96 | C –N stretch | Nitrile |
| 1729.04 | C =O stretch | Aldehyde, ketones |
| 1628.68 | C = O stretch | Carboxylic acid |
| 1470.42 – 1331.46 | N – O stretch | Nitro compounds, aromatics |
| 1246.54 | P−O stretch (strong) | Phosphorus oxide, bonded |
| 1134.60 | C –N stretch (medium-weak) | Aliphatic amines |
| 1041.96 | C – F (stretch) | Alkyl halides |
| 949.32 − 829.66 | =C – H (bending) | Alkenes |
| 764.04 − 665.96 | C− Cl (strong) | Alkyl halides |
| 551.74 | C − Br (strong) | Alkyl halides |

**Table−2: Fourier transform infrared spectrum of palm kernel shell after absorption.**

| Wave number $cm^{-1}$ | Bond Source | Functional group |
|---|---|---|
| 3940.82 −3755.54 | O− H (stretch, free) | Amine, alcohol |
| 3659.04 −3226.72 | **O** −H (stretch, bonded) | Alcohol |
| 3137.94 | **N** −H (stretch) | Amines |
| 3076.18 −2856.16 | **C** − H (stretch) | Alkanes |
| 1624.82 | C = C (stretch) | Alkenes |
| 1408.66 | −C −H (bending) | Alkanes |
| 1254.26 | C –O (stretch) | Alkyl halides |

| | | |
|---|---|---|
| 1103.72 – 1011.08 | C – O (stretch) | Alcohol (strong) |
| 949.32 − 675.26 | = C –H (bending) | Alkenes |
| 574.90 − 517.00 | C −Br (stretch) | Alkyl halides |

The FTIR analysis of the palm kernel shells sample used in gas absorption for the scrubbing of $CO_2$ from bio-methane revealed the presence of alcohols, amine, ketones, aromatics, alkenes, alkanes, carboxyl acids, aldehyde, alkyl halides, phosphorus oxide, nitriles, nitro compounds and alkynes. The most abundant type of bond in this palm kernel samples is the O-H stretch in alcohols with very strong and broad peaks. Comparing functional groups displayed in the FTIR result tables of fresh and spent palm kernels in tables 1 and 2 respectively there is a reduction in the absorption bands (peaks). This reduction was due to the attack on the O-H group by the reaction between NaOH solution and $CO_2$ gas. This means that oxygen has been used up to create more porosity on the palm kernel shell and thereby created more surface area for mass/heat transfer in gas/liquid interface [13]. It could also be noticed that some peaks were shifted or disappeared and some new peaks like alkanes and phosphoric acids were formed too. This change observed in the spectra chart indicated possible involvement of those functional groups in the absorption process.

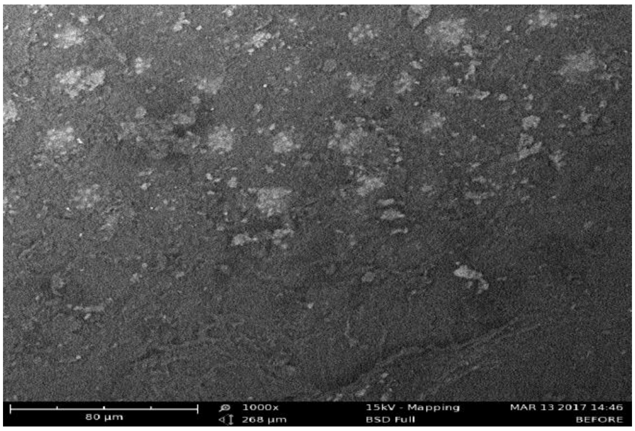



**AP**

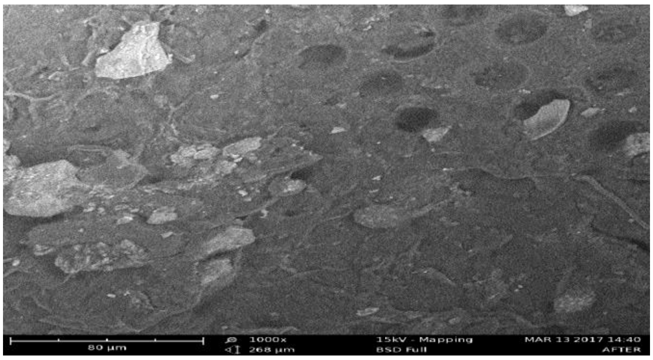
**Fig-1: FTIR chart of palm kernel shell before and after absorption**

**Scanning Electron Microscope (SEM) results**

Figures 2 and 3 showed the SEM analysis of fresh and spent palm kernel shell respectively. The SEM analysis was used to show the morphology or texture and surface topography/ pore structure of the palm kernel shell which are essential for expelling volatiles from the solution. The SEM images in the figures 2 and 3 revealed the porous and heterogeneous structure of the fresh palm kernel shell before and after the absorption process using NaOH solution as the absorbent liquid. The micrographs reveal the micro pores through which the kernel exchanged fluids with the surrounding mesocarp. There was an increase in macro porosity at 15kv mapping and 1000x magnification as observed in the SEM image which was consistent with Okoroigwe et al, 2014 after scrubbing raw bio-methane with NaOH solution. It could be inferred that palm kernel shells use for bio-methane scrubbing serve two purposes- provision of high mass/heat transfer contact between the gas and liquid and expelling of volatiles through its macro pores [12].




**Fig−2: SEM analysis of fresh palm kernel shell before scrubbing process**

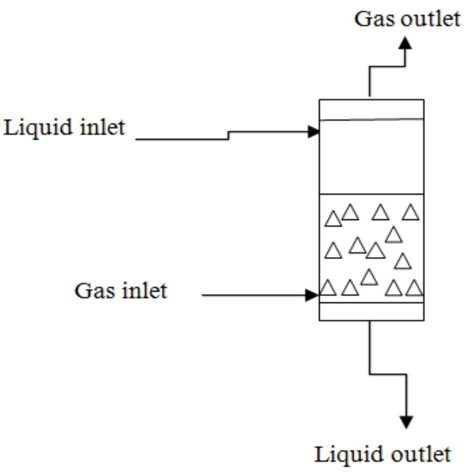



**Fig−3: SEM diagram showing increased macro pores after scrubbing process.**

**Selection of palm kernel as packing materials for the scrubber**

Apart from the economic advantage of palm kernel shell over other packing materials being an agricultural waste product, palm kernel shells were used in the packed column considering the following factors:

- Palm kernel shells were chemically inert to caustic soda attack.
- Pressure drop was lower in packing.
- It contained adequate passages for both gas and liquid streams without excessive pressure drop due to good void fraction of the palm kernel shells.
- It provided good contact between liquid and gas.
- It was the cheapest packing ever got because it is an agricultural waste.




**Fig−4: schematic diagram of biogas scrubber**

## CONCLUSION

Local palm kernel shells can be used as packing materials for the scrubbing process to enhance mass and heat transfer contact between the gas and liquid phases and facilitate exchange of volatiles between the solution and the gas phase. This was because it fulfilled all the requirements of a packing material as proposed by Sinnot 2005 to yield methane gas of natural gas standard without decomposition.

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