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Evaluation of Guinea Corn (*Sorghum bicolor L.*) Chaff for Production of Bioethanol

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Abstract

Guinea corn chaff was pre-treated by screening and ultrasonication processes. Fiber fraction analysis of the untreated and treated chaffs were determined using fibertex analyser. Hydrolysis of the sample was carried out with 4% w/v sulphuric acid and the hydrolysate analysed for monomeric sugars (glucose, xylose, arabinose) using High Performance Liquid Chromatography. The hydrolysed sample was fermented for 5 days to produce bioethanol. Bio-CaO produced from waste fruit was used for purification of the bioethanol produced. Surface morphology and structural composition of the pre and post hydrolysed chaffs were studied. The fuel properties of the bioethanol were carried out and characterised with FTIR. Pre-treatment result in fractional increase in fiber content of 74.96%. Neutral detergent and acid detergent fibers recorded for the untreated and treated chaffs were 74.36%, 74.56%, 29.40% and 29.20% respectively. Hemicellulose content recorded for the untreated and treated chaffs were 44.96% and 45.36%. The result of the hydrolysis using HPLC for monitoring the release of the monomeric sugars were established at 1:5 liquor ratio and 1.21 micrometre particle size. The surface morphology of the sample showed an organised and rupture surface before and after hydrolysis. The fuel properties of the bioethanol conform to the standard values. These results obtained suggests that guinea corn chaffs are good feedstock for bioethanol.

Keywords

Renewable Energy, Biofuel, Bioethanol, Fibertex, Analyser, Monomeric, Hemicellulose Biomass, Fermented

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1. Introduction

Incessant cost of producing petroleum products leads to increase in price of oil in many countries. Negative effects of oil spillage and emission of poisonous gases from fossil fuels have made it imperative to explore alternative energy sources. One of the possibilities of improving the energy sources is the use of plant biomass for bioenergy production [1]. Bio-fuels such as bio-ethanol, bio-diesel and biogas produced from biomass agricultural waste products are increasingly becoming alternative sources of energy globally due to their "replenishability" and environmentally friendly potential [2]. Some of the crops used in producing bioethanol are cassava peels, sugarcane baggasses, millet, cocoyam, sweet potatoes and so on. These crops thrive well in developing countries like Nigeria where agricultural production is basically at the subsistence level.

Sorghum (*Sorghum bicolor*) belongs to the genus Sorghum, tribe Andropogoneae, of the Poaceae family. The species *S. bicolor* includes all cultivated sorghums as well as a group of semi wild and wild plants regarded as weeds [3]. According to Guo, Sorghum bicolor (sweet sorghum) is a high yielding species and it is considered as an energy crop that is able to survive in water limited environments [4]. Harvesting of sweet sorghum can be done three to four times yearly if proper irrigation is applied. Its short life time, soil, climatic

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adaptability and relatively low production cost is considered an advantage in bioethanol production [5]. Among annual energy crops, Serra states that biomass sorghum is considered to be a feedstock that could play an important role in energy production as a dedicated lignocellulosic energy crop [6].

The chaffs after being proccessed serve as feedstock for energy conversion which include bio-oil [7] and syn-gas. The objective of this study was to evaluate the production of bioethanol from guinea corn (*Sorghum bicolor L.*) chaff using acid hydrolysis method. The fiber fraction analysis of chaff, structural composition and surface morphologies of the pre and post-hydrolysed samples were evaluated. Fuel properties of the derived bioethanol produced from the corn chaff are compared with the conventional ethanol.

2. Materials and Methods

2.1. Materials

Guinea corn used for this study was obtained from Odo-Owa farm Kwara State, Nigeria. The chemical reagent used were all analytical grade and purchased from Sigma Aldrich (St. Louis, M. O). Distilled water was use for all washing, cleaning and preparation of solutions. The sample was cleaned to remove dirt, soaked for four days, wet milled with an attrition mill and sieved to remove the starch. The chaff was sun dried for four days, weighed and stored for analysis.

2.2. Analysis of the Chaff

Fiber fraction analysis of the chaff such as neutral detergent fiber, acid detergent fiber, hemicellulose, cellulose and lignin contents were determined with fibertex analyser [8].

2.3. Hydrolysis of the Sample

Acid hydrolysis was carried out using a modified method [9] in a percolating reactor with liquid recirculation. Ten (10) grams of the sample was weighed and hydrolysed at reaction conditions of 4%w/v sulphuric acid, 140°C and 2 h contact time. Monomeric sugars content in the hydrolysate were identified and quantified with HPLC. Structural composition and surface morphology of the pre and post hydrolysed samples were studied using Fourier Transform Infra-red and Scanning Electron Microscopy.

Fermentation of the Hydrolysate with Yeast (Saccharomyces cerevisea) from Rotten Fruits

Hydrolysed sample was fermented [10] by inoculating a cell of 48 h old and incubating while shaking at 200 rpm. The sample was inoculated into the fermentation medium after 18 h and left for 5 days. The fermented broth was distilled at 78°C using a simple distillation set-up.

2.4. Production of Calcined Waste Cow Bone

Cow bone ash (CBA) used was produced from locally obtained cow bones waste. The bones were washed in clean water to remove any dirt present, sun dried, crushed (using electrical bone crushing machine) and ground into small particles size. The small particles obtained was calcined at temperature of 800°C in a muffle furnace for 4 h to ensure complete transformation of calcium compound into apatite [11]. The catalyst produced after calcination was characterized with X-ray diffractometer (JEOL, JDX-3530, 2kW, Tokyo, Japan) at a scan rate of 12° min^{-1} from 5° to 50°. It was poured unto a glass plate and inserted into its space in the XRD machine. The sample show result on composition of calcium oxide from different calcined temperature.

2.5. Purification of the Fermented Broth

The liquor was further purified by refluxing with bio-calcium oxide catalyst. A 2.5 g of catalyst was added to 30 ml of the liquor, reflux for 3 h and the bioethanol was distilled with simple distillation apparatus. Then redistilled with another distillation set-up consisting of a Liebig condenser and fractionating column. Sodium sulphate was added as a dehydrating agent and filtered. Fuel properties of the bioethanol was carried [12].

2.6. Spectroscopic Analysis

The chaff, before and after hydrolysis and the ethanol distillate were characterised using Fourier Transform Infrared Spectroscopy (FTIR).

3. Result and Discussion

3.1. Fiber Fraction Analysis

Fiber is the organic portion of the feeds which is not easily digestible. Neutral detergent fiber represent the insoluble fiber in feeds that is slowly digested. The result of the fiber fraction in Table 1 showed that neutral detergent fiber recorded for untreated, pre-treated and sonicated guinea corn chaff were (74.36, 74.56, 74.96%) respectively. This is an indication that total cellulosic material was present more in sonicated chaff at 74.96 than the others. The recorded values were higher than (53.51, 57.38 and 58.12%) reported for dried distiller grains [9]. The higher neutral detergent fiber recorded leads to an increase in cellulose and hemicellulose resulting into higher yield of monomeric sugars. Acid detergent fiber represents the fibrous, least-digestible portion of roughage which include cellulose, lignin and silica. The values recorded for untreated, pre-treated and sonicated guinea chaff were (29.40, 29.20, 29.30%) respectively. These

results were consistent with (27.76, 26.57 and 28.39%) reported for dried distiller grains [9].

Hemicellulose represent the non-digestible part of the fiber. The values recorded for hemicellulose content of the untreated, pre-treated and sonicated maize chaff were (44.96, 45.36, 45.66%) respectively. They were higher than (25.75, 30.81 and 29.73%) reported for dried distillery grains [9]. However, the high hemicellulose and neutral detergent fiber content recorded in this research work shows that guinea chaff is a promising feedstock for bioethanol production [9].

Table 1. Fiber fraction analysis of the chaff.

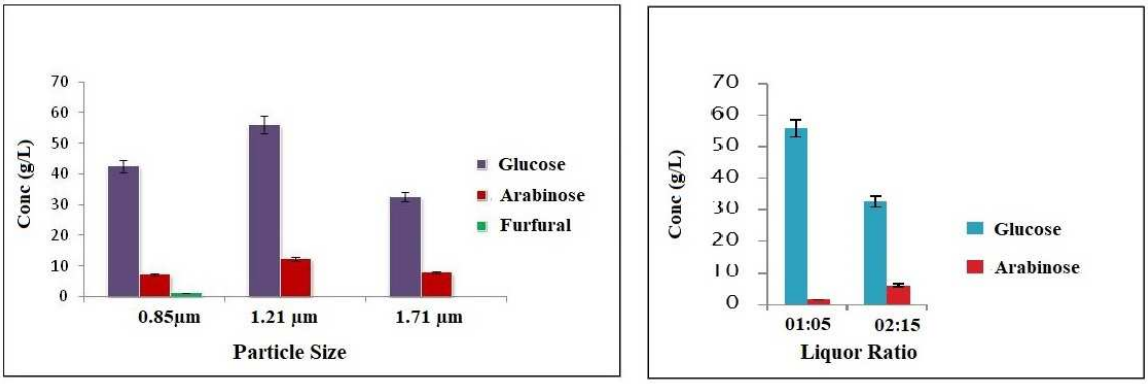
Analysis (%)	Untreated guinea corn chaff	Pretreated Screened guinea corn chaff	Screened and sonicated guinea corn chaff
Neutral detergent fiber	74.36%	74.56%	74.96%
Acid detergent fiber	29.40%	29.20%	29.30%
Hemicellulose	44.96%	45.36%	45.66%

Means of the same letter are not significantly different from each other ($p \leq 0.05$)

3.2. Hydrolysis Parameters

The result in Figure 1a shows the effect of particle size on sugar level of the guinea corn chaff hydrolysate using HPLC at 4% w/v acid concentration, temperature of 120°C, 120 min. The particle size 1.21 µm was the best for the release of the monomeric sugars (glucose at 56.04 and arabinose at 12.28 while xylose was not detected). The only degradation product detected in the result was furfural at 0.85 µm which was relatively lower in other particle size. The experimental condition was unfavourable for the release of hydroxylmethylfurfural which was not discerned. Moreover, research findings by Ivone on the effect of particle size using dilute acid hydrolysis on giant reed (*Arundo donax*) show a selective hydrolysis of the hemicellulose regardless of the particle size fractions assayed [13].

Figure 1b shows the effect of biomass to liquor ratio on sugar level of guinea corn hydrolysate. The result show that as biomass ratio increased the sugar level decreased from 1:05 to 2:15 for glucose while a different trend was observed for arabinose. Glucose decreased from 56.04 to 32.56 g/L while a slight increase was observed in arabinose. The best liquor ratio favourable for the release of monomeric sugar was 1:05 and there was no degradation product detected. Similar research by Tune on liquor ratio reported that decrease in the monomeric sugar concentration with higher liquor ratio could be due to decrease in acidity of the hydrolysis medium with increasing liquor ratio [14].



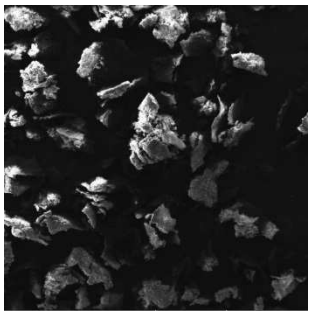
a. Effect of particle size on sugar level

b. Effect of biomass to liquor ratio on sugar level

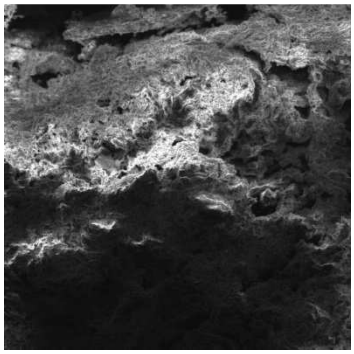
Figure 1. Effect of particle size and biomass to liquor ratio on sugar level.

3.3. Surface Morphology

The results in Plates 2a and b show the surface morphologies of the pre and post-hydrolysed samples. The morphology of the sample before hydrolysis shows an organised structure compared with the rupture surface after hydrolysis which implies that the hydrolysis process was effective. Dania reported that SEM micrographs demonstrate changes in fiber morphology as the severity of the hydrolysis conditions while working on dried distiller grains [9].



a. SEM of pre-hydrolysed samples.



b. SEM of post-hydrolysed samples

Figure 2. SEM of pre and post-hydrolysed samples.

3.4. Fourier Transform Infrared Spectrophotometer (FTIR) Analysis of the Chaff

The result of FTIR analysis in Figure 3, show combined spectra of the samples before and after hydrolysis. The structural composition of the functional groups in the samples show O-H group absorption peak around 3350 cm^{-1} which was more broad and intense in post-hydrolysed samples than in pre-hydrolysed. C-H stretching vibrations of CHO two bands around 2900 cm^{-1} are more pronounced upon hydrolysis. The peak around 1720 cm^{-1} indicates C=O which was only found in pre-hydrolysed samples. C-H vibration of aromatic ring absorption peak around 1600, 1500 and 1450 cm^{-1} had different intensities for both pre-hydrolysed and post-hydrolysed samples. C-H bending vibration of $C\text{-}CH_3$ absorption peak around 1380 and 1360 cm^{-1} was more intense in post-hydrolysed than pre-hydrolysed samples. C-O-C ketone absorption peak with great intensity around 1000-1100 cm^{-1} was more pronounced in post-hydrolysed samples.

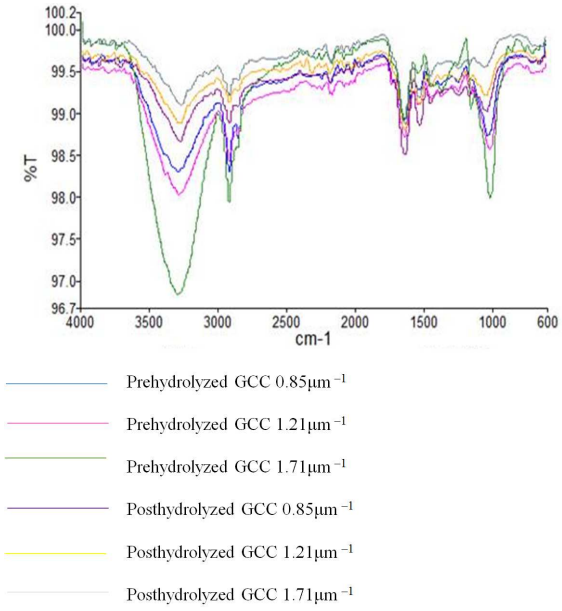


Figure 3. Combine FTIR spectra of pre and post-hydrolysed guinea corn chaff.

3.5. Fuel Properties of the Derived Bioethanol

Table 2 show fuel properties of the conventional ethanol and derived bioethanol produced from the corn chaffs. The results show that specific gravity of derived bioethanol from guinea corn chaff was 0.833 compared favourably with the standard ethanol value of 0.7974. The refractive index value of 1.3614 compared favourably with the standard value of 1.3600 [15] as well as the value reported for cocoyam (*Colocasia Antiquorium*) at 1.3607 [16]. The flash point for guinea chaff derived ethanol recorded as 44 was higher than the standard ethanol with a value of 38. The alcohol by volume recorded at 87 was quite lower comparable with the standard ethanol. Also, the calorific valves of 568.14 was recorded for the sample. The value was quite comparable with the standard ethanol value of 567.03 [17]. The result obtained shows that the sample is a promising feedstock suitable for bioethanol production.

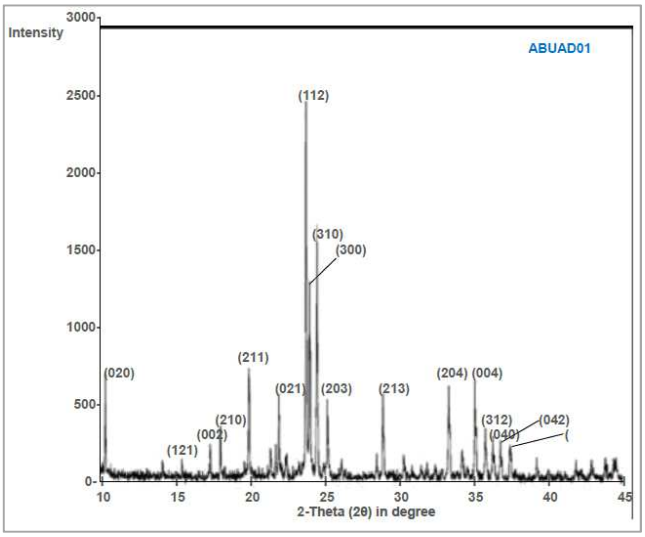


Figure 4. XRD pattern of the ash obtained by calcined cow bone.

3.6. X-ray Diffraction Analysis

Figure 4 shows X-ray Diffraction pattern of the ash obtained through calcination of cow bone**.** The sharp peak shows that the material was well cystallized. Prominent peak was seen at 2-Theta equal to 112 which corresponds to the presence of calcium oxide (CaO), when compared with standard peak patterns of Joint Commitee on Powder Diffraction Standards file. This result show that the powder cow bone ash was converted to calcium oxide after calcination.

3.7. Fourier Transform Infrared Spectrophotometer (FTIR) Analysis of Bioethanol

Figures 5 and 6 show the FTIR analysis of crude and purified bioethanol generated from guinea corn chaffs. Figure 5

shows O – H stretch of alcohol broad absorption peak for crude bioethanol in the region 3336.00 cm^{-1} indicating the presence of water, hence the needs for purification. Other absorption peak in the spectra which were present due to impurities are C=O carbonyl around 1635-1642 cm^{-1} and peak around 330-650 cm^{-1}. Figure 6, shows a change in the O – H absorption peak that appeared after purification process at 3336.36 cm^{-1} which was not wide and broad as they were seen in the crude bioethanol. The result shows the effectiveness of the purification process which has remove the water presence in the sample. However, other absorption peaks seen in the spectra were C – H stretching vibration of CH_3 at 2902.28 cm^{-1} and C – O stretch of alcohol at two band in the region of 1086.67 cm^{-1} (1044.66 cm^{-1}). Khushna investigated that the FTIR spectrum of bioethanol from banana peel showed characteristic peaks at 3404.01 cm^{-1} (– OH bonds); 2933.54 cm^{-1} (– C – H bonds); and 1011.06 cm^{-1} (– C – O bonds). However, the peaks in this study were consistent with the one reported for banana peel pulp [18].

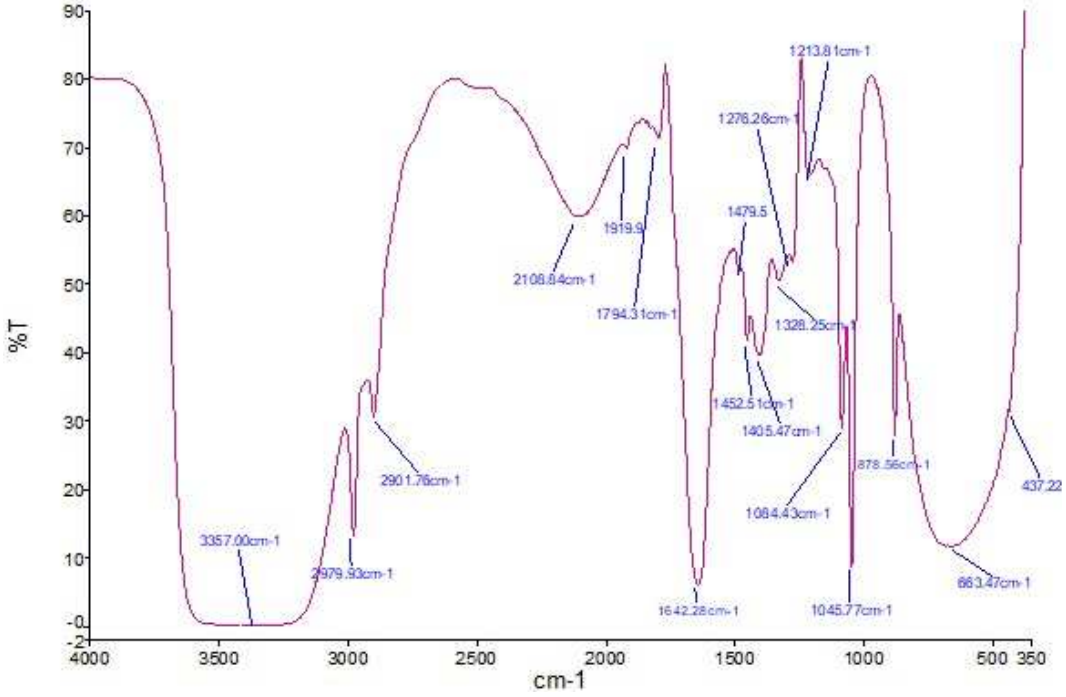


Figure 5. FT-IR spectrum of crude bioethanol from guinea corn chaff.

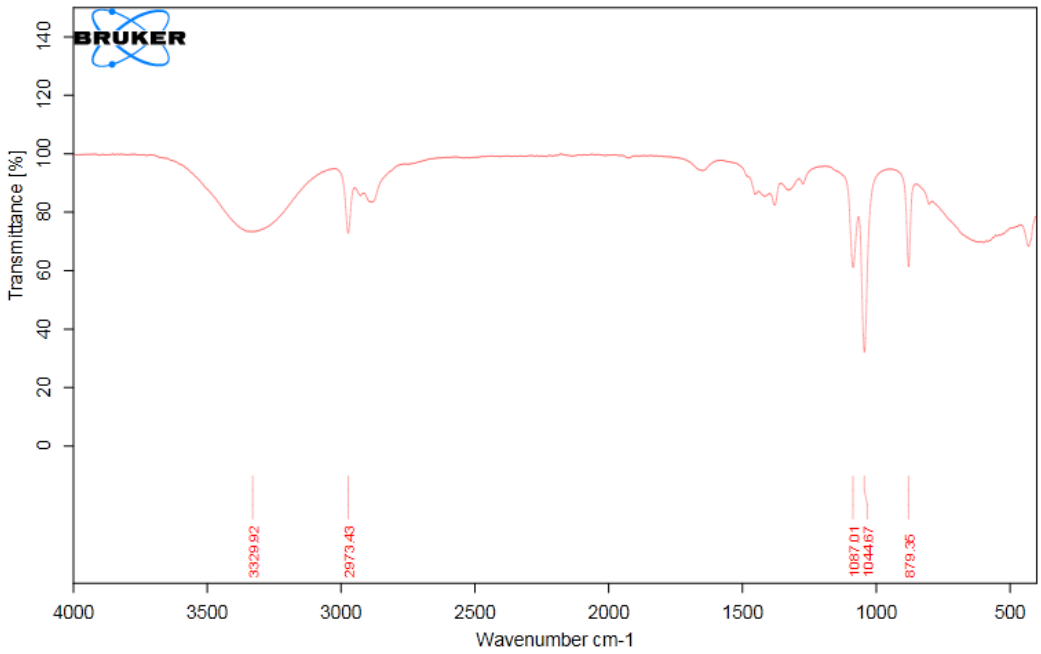


Figure 6. FT-IR spectrum of purified bioethanol from guinea corn chaff.

Table 2. Fuel properties of Conventional Ethanol and Derived Bioethanol.

Properties	Standard Conventional ethanol	Bioethanol from guinea corn chaff
Chemical formula	C_2H_5OH	C_2H_5OH
Boiling point in°C	78	78
Specific gravity at 30°C	0.7974 ± 0.01	0.833 ± 0.02
Alcohol by volume (%)	98 ± 0.00	86 ± 0.01
Refractive Index	1.3600 ± 0.01	1.3614 ± 0.01
Flash point°C	38 ± 0.01	44 ± 0.02
Calorific Value in J/g^{-1}	567.03 ± 0.00	568.14 ± 0.02
Percentage Yield	89	

The result is mean ±standard deviation

4. Conclusion

This study confirmed the suitability of guinea corn chaff as potential feedstock for bioethanol considering the fiber fraction analysis which signalled an increase in cellulose and hemicellulose contents with resultant higher yields of monomeric sugar. The chaff contained high fermentable sugar which after being pre-treated opens the pore size of the chaff for effective hydrolysis. The properties of the fuel derived ethanol indicate that it has a larger density than the conventional ethanol based on the value of the specific gravity though it is still less dense than water. Flashpoint value is within range of the conventional ethanol also which makes it a promising candidate as a biofuel alternative. However, the result also confirmed that crude bioethanol produced can be purified with bio-CaO. The optimum conditions for effective release of the sugars were established at 1:5 liquor ratio and 1.21 µm particle size. The research findings confirmed that guinea corn chaff is a potential feedstock for the production of bioethanol.

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