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Extraction and Transesterification of cottonseed oil

[1]Selma .A.Hussein , [2]Ehsas. A. Mohammed,[3] Gurashi. A.Gasmelseed,

[1]Industrial& Systems Engineering Dept, College of Engineering, Princess Nourah bint Abdulrahman University, Sudan university of science and technology, sahussein@pnu.edu.sa.

[2]College of Engineering ,University of Karry,ehsaselfatih@gmail.com.

[3]College of Engineering , Eltigana University, gurashigasm@gmail.com.

Abstract

The amount of oils in cotton seed was determined by the physical extraction method, and the percentage of free fatty acid in the oil was calculated. Extraction and transesterification experiments were conducted in the Sudan Research and Consultancy Center (RCC), Khartoum-Sudan. The percentage of oil content in the sample was 30.09% based on a wet basis. The crude cottonseed oil was used as a raw material for biodiesel production because its demands as an edible oil has been reduced and due to the shortage in fuel oil in Sudan. The transesterification reaction was carried out with a methanol/oil ratio of (1:6-1:12mol/mol), catalyst of (0.003) w%, temperature (of 65 ^{0}C), different reaction times of (10-30mints), and mixing rate of (500 rpm). The sample of biodiesel was introduced into the GC-Ms, and the yield was found to be (99.3w%). The properties of the biodiesel produced including density (0.8960 g/cc), kinematic viscosity (7.702cSt), specific gravity (0.8960), and calorific value (43.3Mj/kg) were determined and compared with the international standard specifications and found to be in agreement.
Keywords: Cottonseed oil, Extraction, Transeserification, Biodiesel.

الملخص:
تم تحديد كمية الزيوت في بذور القطن بطريقة الاستخراج الفيزيائي ، وتم حساب نسبة الأحماض الدهنية الحرة في الزيت. أجريت تجارب الاستخراج والأسترة التبادلية في مركز السودان للبحوث والاستشارات (RCC) ، الخرطوم - السودان. كانت نسبة محتوى الزيت في العينة ٣٠،٠٩٪ على أساس رطب. تم استخدام زيت بذرة القطن الخام كمادة خام لإنتاج وقود الديزل الحيوي بسبب انخفاض الطلب عليه كزيت صالح للأكل وبسبب نقص زيت الوقود في السودان. تم إجراء تفاعل الأسترة التبادلية بنسبة ميثانول / زيت بنسبة (١: ٦-١: ١٢ مول / مول) ، محفز (٠،٠٠٣)٪ وزن ، درجة حرارة (٦٥ درجة مئوية) ، أوقات تفاعل مختلفة (١٠-٣٠ دقيقة) ، ومعدل خلط (٥٠٠ دورة في الدقيقة). تم إدخال عينة الديزل الحيوي في GC-Ms ، ووجد أن الناتج كان (٩٩،٣٪ بالوزن). تم تحديد خصائص وقود الديزل الحيوي المنتج بما في ذلك الكثافة (٠،٨٩٦٠ جم / سم مكعب) ، واللزوجة الحركية (cSt٧،٧٠٢) ، والجاذبية النوعية (٠،٨٩٦٠) ، والقيمة الحرارية (٤٣،٣ ميجا جول / كجم) وعند مقارنتها بالمواصفات القياسية الدولية ووجد أنها مطابقة للمواصفات.

الكلمات المفتاحية :- زيت القطن، الاستخلاص ، الاسترة، الوقود الحيوي.

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Introduction:-

Cotton belongs to the Malvaceae family. It is an important crop that yields the natural fibers used by the textile industry. Cottonseed oil is extracted from the seeds of the cotton plant after the removal of cotton lintiles. Cotton oil is used in the industries for soap, and glycol as well as being an edible oil. In addition cottonseed oil is a suitable raw material for biodiesel production. There are two types of processes for the production of vegetable oils. One is mechanical while the other is solvent extraction. In the mechanical method, the oil is extracted by pressing the oil seeds. In the solvent extraction, solvents are used for extracting the oil, and the solvent is recovered and recycled, (6). Biodiesel is usually obtained by the transesterification of oils or fats by reacting a short-chain alcohol, such as methanol, in the presence of a homogeneous base-catalyst (typically NaOH). The transesterification reaction mainly depends on the nature of the feedstock, catalyst concentration, alcohol-oil molar ratio, temperature, agitation speed, pressure, and residence time as well as moisture content and amount of tree fatty acids in the transesterification reaction, one mole of triglyceride reacts with three moles of alcohol to form one mole of glycerol and three moles of the respective fatty acid. After the reaction, the mixture forms two phases: the glycerin-rich-phase and ethyl esters-rich-phase. The two phases can be separated by gravity settlement.(6)

Objectives:-

1- Extraction and separation of cottonseed oil.
2- Production of biodiesel from cottonseed oil.

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Literature review:

The production of cottonseed methyl ester (biodiesel) from non-edible vegetable oils for a diesel substitute is particularly important because of the decreasing trend of economical oil reserves, environmental problems caused due to fossil fuel use, and the high price of petroleum products in the international market. The present work presents an optimized protocol for the production of methyl ester through the alkaline-catalyzed transesterification of cottonseed oil. Three principal variables, namely the molar ratio of methanol to oil, amount of catalyst, and reaction temperature affecting the yield of the alkaline-catalyzed production of methyl ester from cottonseed oil, were investigated.

In the previous work it was investigated that the Smaller the particle size (450μm), the higher the mass transite-rate.

Invegistigation of environmental impact of utilization of cotton seed oil blend with gasoline starting its impact from planting, irrigations and solid waste incerenareon. In other countries, the planting of cotton is mainly for cotton fibres for texile industry and that the cotton Lintiles are used for Nitrocelluse Production as solvenls, but the seed are a byproduct which is subjected to leaching of oil for edible uses. Due to poor quality for cotton seed oil as edible oil, and of environmental protection and green house effect, cotton seed oil can be esterified to biodiesel.

Material and methods:-

Apparatus and materials:-

The apparatuses used were (a soxhlet apparatus, gas chromatography mass spectrometerie (GC/MS), stirrer, and separating funnels), and the chemicals and materials used were (cottonseed, cottonseed oil, n-hexane, methanol, sodium hydroxide).

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Methods:-

Extraction:-

The cotton seed was roastedand peeled and then well grinded. A sample of weight of 8.93g was submerged in 250 ml of n-hexane and placed in a Soxhlet apparatus for 4 hours. The sample was extracted, dried, and weighted, and the oil content of the seed was calculated:

$$\text{Oil content} = \frac{w\ of\ oil}{w\ of\ sample} * 100 \ldots\ldots\ldots\ldots \quad (1)$$

Free Fatty Acid Test (F.F.A):

A sample of cottonseed oil weighting 2g was dissolved in 30 ml of ethanol and titrated with 4.69 ml of NaOH (0.01M) to ensure that there was no acid in the solution. Then a phenonaphthalein detector was added, and the sample was titrated with NaOH(0.01M).

$$\text{F.F.A. \%} = \frac{volume\ of\ NaOH * M\ of\ NaOH * 28.2\%\ as\ olic}{w\ of\ oil} \ldots\ldots\ldots \quad (2)$$

4.2.3- Transesterification process:-

In this study, nine experiments were conducted to produce biodiesel in a batch-scale transesterification process using crude cottonseed oil.

A known quantity of crude cottonseed oil was weighed and transferred into a 250ml conical flask and preheated at 60^0C in a water bath for 30 min. A quantity of sodium hydroxide pellets was weighted and dissolved into the desired amount of methanol and allowed to dissolve completely. The quantities of oil, methanol, and sodium hydroxide were weighed according to the values provided in the experimental design, the dissolved methanol-sodium hydroxide mixture (sodium methoxide)was added to the hot oil and mixed with a stirrer. The reaction temperature and time were regulated based on the experimental design objective. The reaction was stopped at the end of each specific time, and the product was transferred into the separating funnel

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And allowed to stand overnight for proper sedimentation. The glycerol was then drained the following day from the separating funnel as it is heavier than the biodiesel. The procedure was repeated 9 times corresponding to the total number of experiments generated by the CCD design. The percentage yield of biodiesel for each experimental run was evaluated by relating the weight of crude cottonseed oil consumed by the reaction using the following equation:-

$$\%\text{yield} = \frac{weight\ of\ biodiesel\ produced(g)}{weight\ of\ oil\ used\ (g)} * 100 \ldots\ldots\ldots \quad (3)$$

GC-MS Analysis of Transesterified Oil (FAME):-

The composition of the biodiesel was examined through a gas chromatographic mass spectrometric analysis. Samples (1.0µl) were injected in a split mode (split/column flow ratio 50:1).the column head pressure of the carrier gas (helium) was 13.332 psi, the injection temperature was 280^0C. and the total run time was 27.5 min. The GC-MS apparatus was connected to a PC to run the software for data acquisition and processing.

Characterization of Biodiesel:-

Fuel property tests were conducted for kinematic viscosity at 50^0C (ASTM D4052). Specific gravity (ASTM D4052),density@ at 15^0C (ASTM D4052), and gross alorific value according to Sudan Standards for the trans esterified oil obtained from cottonseed.

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Results and Discussion:-

Extraction and F.F.A:-

The average oil content of the cottonseed is approximately (14%-28%). In this study was found that the oil content in the cottonseed was (30.09%), and the free fatty acid in the oil was (0.58%).

Transeserified Oil Yield:-

In this study a minimal amount of methanol was used, based on previous studies. In the experimental work, 9 experiments used 2.8g of sodium hydroxide dissolved in 100ml of methanol, and then different volumes of cottonseed oil were added. The mlar ratio, was (1:6, 1:8 and 1:12 methanol/oil), the times were (10 -30min), and the rate of mixing was 500 rpm. The temperature was 65^0C , and the samples were separated after 12 hours, as shown in Table (1

Table(1): the yield of transesterified oil,

Molar ratio	Time/min	Yield%
16	10	87.38
	20	92.79
	30	94.40
18	10	97.13
	20	98.02
	30	98.47
1.12	10	99.03
	20	99.11
	30	99.42

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Characterization of Biodiesel:-

The fuel properties such as kinematic viscosity, specific density, and calorific value determined by ASTM standard methods, and the results are shown in Table(2).

Table (2) Biodiesel analysis:

Test Name	Value measured
density@ 15^0C (ASTM D4052)	0.8960 g/cm^3
S.G (ASTM D4052)	0.8960
Kinematic viscosity @ 15^0C (ASTM D445)	7.7020 c5t
Calorific value gross (Calc)	43.9670MjJ/kg

GC-MS Analysis:-

In this study, the biodiesel from the transesterification reaction was analyzed by GC-MS, and the results are shown in table (3)

Table(3) GC-MS characterization of Transesterified Oil obtained from Cottonseed.

Test Name	Test Method	Unit	Result
GC-MS	GC-MS		
Methyl tetradecanoate	GC-MS	Wt%	0.744
Methyl hexadec-9-enote	GC-MS	Wt%	0.483
Methyl hexadecanoate	GC-MS	Wt%	25.405
Methyl octadec-9,12-dicnoate	GC-MS	Wt%	50.913
Methyl octadec -9-enoate(E)	GC-MS	Wt%	18.500
Methyl octadec-9-enoate(Z)	GC-MS	Wt%	0.952
Methyl octadecanoatcte	GC-MS	Wt%	3.004

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Conclusions:-

In this study, cottonseed oil was extracted to a Soxhlet apparatus using n-hexane, and the oil content in the seed was found to be 30.09%. the F.F.A. in the oil was calculated and found to be to 0.58%. The possibility of producing biofuels using methanol as limiting reactant was also investigated. Chemical and physical properties and the calorific value of the biodiesel produced were also measured. A control strategy was developed and analyzed.

Recommendations:-

1. Improvement of cottonseed oil for edible uses.
2. Economics of cottonseed edible oil copared with biodiesel from the same.
3. Development of digital control system for biodiesel plant.

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