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BIODIESEL PRODUCTION FROM COTTONSEED SOAPSTOCK ACID OIL VIA TWO-STEP ACID ESTERIFICATION AND ALKALINE TRANSESTERIFICATION

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Abstract. Soapstock, the main by-product of alkali refining of cottonseed oil, is a low-cost lipid feedstock whose high free fatty acid (FFA) content prevents its direct use in conventional alkali-catalysed biodiesel processes. In this study, acid oil recovered from cottonseed soapstock by sulphuric acid acidulation (acid value 118.6 mg KOH/g, FFA $\approx$ 59.7 %) was converted into fatty acid methyl esters (FAME) by a two-step process. In the first step, FFAs were esterified with methanol over H₂SO₄; the effects of methanol and catalyst loadings were examined at 65 °C for 120 min. A methanol loading of 40 wt% and 2 wt% H₂SO₄ reduced the acid value to 1.7 mg KOH/g (98.6 % FFA conversion). In the second step, the pretreated oil was transesterified with methanol (6:1 molar ratio) using KOH at 60 °C for 60 min, and the crude esters were purified by vacuum distillation to remove gossypol-derived pigments and unsaponifiable matter. The overall yield of distilled FAME was 76.8 % relative to acid oil. The product had an ester content of 97.6 %, kinematic viscosity of 4.31 mm²/s and acid value of 0.38 mg KOH/g, meeting the main EN 14214 and ASTM D6751 limits, while its oxidation stability (4.9 h) requires antioxidant addition. The results show that cottonseed soapstock, abundant in Uzbekistan, can serve as a viable feedstock for biodiesel production.

Keywords: biodiesel; cottonseed oil; soapstock; acid oil; free fatty acids; esterification; transesterification; vacuum distillation



1. Introduction

Biodiesel, defined as mono-alkyl esters of long-chain fatty acids, is a renewable, biodegradable and practically sulphur-free substitute for petroleum diesel [1, 2]. Its large-scale use is limited mainly by cost: refined vegetable oils account for the largest share of biodiesel production costs, so low-value lipids are increasingly considered as alternative feedstocks [3, 4]. Among them, soapstock – the alkaline by-product of edible-oil refining – is attractive because it is generated in large quantities at existing oil mills and has little market value [4].

Uzbekistan is one of the largest cotton producers in Central Asia, and cottonseed oil is the principal edible oil processed by domestic oil-and-fat plants. Alkali refining of crude cottonseed oil is accompanied by relatively high refining losses, because the soapstock carries not only neutralised fatty acids but also gossypol and other pigments [5]. Converting this stream into biodiesel would therefore add value to the domestic oil industry and reduce waste.

The key technical obstacle is the high FFA content of acid oil obtained from soapstock. In alkali-catalysed transesterification, FFAs react with the catalyst to form soaps, which consume the catalyst, form emulsions and hinder glycerol separation; for this reason, an FFA content below about 1 % is generally recommended [6]. Two-step processes, in which FFAs are first esterified over an acid catalyst and the remaining triglycerides are then transesterified with an alkaline catalyst, have been shown to be effective for high-FFA feedstocks such as yellow grease and used frying oils [6–8]. Haas [4] demonstrated the feasibility of producing biodiesel from vegetable oil soapstock, and Keskin et al. [9] tested cotton oil soapstock biodiesel blends in a diesel engine. However, systematic data on processing conditions for acid oil from cottonseed soapstock of Central Asian origin, and on the purification needed to remove gossypol-derived colour bodies, remain limited.

The aim of this work was therefore to (i) characterise acid oil recovered from cottonseed soapstock of a local oil plant, (ii) determine suitable conditions for acid-



catalysed pre-esterification, and (iii) evaluate the quality of the final FAME obtained by alkaline transesterification and vacuum distillation against EN 14214 and ASTM D6751.

2. Materials and methods

2.1. Feedstock and acidulation

Cottonseed soapstock was obtained from the refining shop of “...” oil-and-fat plant (Namangan region, Uzbekistan). Acid oil was recovered by acidulating the soapstock with 20 % H₂SO₄ at 85–90 °C to pH 2–3 under stirring, followed by settling, separation of the aqueous phase and washing of the oil layer with hot water. The acid oil was dried under vacuum at 105 °C. Methanol (99.5 %), sulphuric acid (96 %) and potassium hydroxide (analytical grade) were used as received.

2.2. Acid-catalysed esterification (step 1)

Esterification was carried out in a 500 mL three-neck flask equipped with a reflux condenser, mechanical stirrer (600 rpm) and thermometer, placed in a thermostatic bath. Methanol (20–50 wt% of acid oil) containing dissolved H₂SO₄ (1–3 wt% of acid oil) was added to the preheated acid oil, and the reaction was conducted at 65 °C for 120 min. After the reaction, the mixture was allowed to settle, the upper methanol–water phase was removed, and the oil phase was washed and dried. FFA conversion was calculated as $X = (AV_0 - AV)/AV_0 \times 100 \%$, where AV_0 and AV are the acid values before and after esterification.

2.3. Alkaline transesterification (step 2)

The pretreated oil was transesterified with methanol at a methanol-to-oil molar ratio of 6:1 using 1.0 wt% KOH plus the stoichiometric amount needed to neutralise the residual acidity, at 60 °C for 60 min. After separation of glycerol, excess methanol was evaporated and the ester phase was washed with warm water to neutrality and dried.

2.4. Vacuum distillation

Because crude esters from cottonseed soapstock are dark due to gossypol-derived pigments, they were distilled under reduced pressure (≈ 0.5 kPa, vapour temperature 180–



220 °C). The distillate was collected as the final product, and the dark residue containing pigments, unsaponifiable matter and unreacted glycerides was weighed.

2.5. Analytical methods

Acid value was determined according to EN 14104, ester content by gas chromatography (EN 14103), density (EN ISO 12185), kinematic viscosity at 40 °C (EN ISO 3104), flash point (EN ISO 3679), water content (EN ISO 12937), free and total glycerol (EN 14105), sulphur (EN ISO 20846) and oxidation stability at 110 °C (EN 14112, Rancimat). Moisture and volatiles and unsaponifiable matter of acid oil were determined by AOCS official methods. All experiments were performed in triplicate, and mean values are reported.

3. Results and discussion

3.1. Characteristics of acid oil

The main properties of the acid oil are given in Table 1. With an FFA content of about 60 %, the material is far above the ~1 % threshold acceptable for direct alkaline transesterification [6], which confirms the need for acid pre-esterification.

Table 1. Properties of acid oil recovered from cottonseed soapstock

Property	Value
Acid value, mg KOH/g	118.6 ± 1.4
FFA (as oleic acid), %	59.7
Saponification value, mg KOH/g	192.4
Moisture and volatiles, %	1.2
Unsaponifiable matter, %	3.8
Colour	dark brown

3.2. Acid-catalysed esterification

Table 2 shows the influence of methanol and H₂SO₄ loadings on the residual acid value. Increasing the methanol loading from 20 to 40 wt% at 2 wt% H₂SO₄ markedly



improved FFA conversion, since excess alcohol shifts the esterification equilibrium towards ester formation and dilutes the water produced. Further increase to 50 wt% gave no additional benefit. Raising the catalyst loading from 2 to 3 wt% had only a marginal effect while increasing the amount of acid to be neutralised later. Run 4 (40 wt% methanol, 2 wt% H₂SO₄) was therefore selected, giving an acid value of 1.7 mg KOH/g (FFA < 1 %), which is consistent with the threshold proposed by Canakci and Van Gerpen [6].

Table 2. Effect of methanol and H₂SO₄ loadings on esterification of acid oil (65 °C, 120 min)

Run	Methanol, wt%	H ₂ SO ₄ , wt%	Acid value after step 1, mg KOH/g	FFA conversion, %
1	20	1	14.8	87.5
2	30	1	8.9	92.5
3	30	2	3.6	97.0
4	40	2	1.7	98.6
5	40	3	1.5	98.7
6	50	2	1.6	98.7

3.3. Transesterification, purification and fuel properties

Alkaline transesterification of the pretreated oil proceeded without emulsion formation, and phase separation was complete within 2 h. The yield of crude esters was 81.4 % and that of distilled FAME 76.8 % relative to acid oil; the distillation residue amounted to $\approx$ 5 %. Vacuum distillation converted the dark-brown crude product into a light-yellow liquid, indicating effective removal of gossypol-derived pigments. The properties of the distilled FAME are summarised in Table 3.

Table 3. Properties of distilled FAME from cottonseed soapstock acid oil

Property	FAME	EN 14214	ASTM D6751
Ester content, wt%	97.6	$\geq$ 96.5	—



Property	FAME	EN 14214	ASTM D6751
Density at 15 °C, kg/m ³	881	860–900	–
Kinematic viscosity at 40 °C, mm ² /s	4.31	3.5–5.0	1.9–6.0
Flash point, °C	158	≥ 101	≥ 93
Acid value, mg KOH/g	0.38	≤ 0.50	≤ 0.50
Water content, mg/kg	290	≤ 500	–
Free glycerol, wt%	0.010	≤ 0.02	≤ 0.020
Total glycerol, wt%	0.16	≤ 0.25	≤ 0.240
Sulphur, mg/kg	6	≤ 10	≤ 15 (S15)
Oxidation stability at 110 °C, h	4.9	≥ 8	≥ 3

All measured properties except oxidation stability comply with EN 14214. The slightly higher acid value compared with FAME from refined oil reflects the small amount of FFA released during distillation and storage, but remains within the specification. The low oxidation stability is typical of cottonseed-derived esters, which contain about half linoleic acid [5, 10]; it may be further reduced by the removal of natural tocopherols during refining and distillation. Addition of synthetic antioxidants such as TBHQ is therefore recommended. Overall, the results agree with earlier findings that two-step processing makes high-FFA by-products suitable for biodiesel production [4, 6, 7].

4. Conclusions

Acid oil recovered from cottonseed soapstock (FFA $\approx$ 60 %) was successfully converted into biodiesel by a two-step process. Acid-catalysed esterification with 40 wt% methanol and 2 wt% H₂SO₄ at 65 °C for 120 min reduced the acid value to 1.7 mg KOH/g, enabling subsequent alkaline transesterification without soap formation problems. Vacuum distillation effectively removed gossypol-derived pigments and yielded FAME (76.8 % overall yield) that meets the main requirements of EN 14214 and ASTM D6751, with the exception of oxidation stability, which must be improved by antioxidants. Cottonseed soapstock can thus be considered a promising low-cost feedstock for biodiesel production



in Uzbekistan. Future work will address process economics, methanol recovery and the use of heterogeneous catalysts.

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