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Preparation and Characterization of Biodegradable Diethanolamide Surfactant from Cotton Seed Oil

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Abstract

The heavy reliance of cosmetics, paint, and other manufacturing industries on edible plant-based oils has driven up the cost of these oils, leading to a significant imbalance between their domestic and industrial usage. With a projected population surge on the horizon, competition for these oils between domestic and industrial sectors could escalate, potentially creating an unsustainable situation for consumers. To address this challenge, this research explores and characterizes biosurfactants derived from cottonseed oil as an alternative fixed oil source. This renewable source shows promise as a commercial raw material for feedstock and oil-based industries, aligning with Sustainable Development Goal 11 (SDG 11) on sustainable production. In this study, diethanolamide biosurfactant was prepared from cottonseed oil using established analytical methods. The modified product was characterize using UV-Visible and Fourier Transform – Infrared (FTIR) Spectroscopy.

Keyword: biosurfactants, diethanolamide, cottonseed oil, cosmetics, oil-based industries

1. Introduction

Surfactants are wetting agents that lower the surface tension of a liquid and can also lower the interfacial tension between two liquids, allowing for easier spreading. A surfactant molecule is made up of two components that have differing solvent affinity. One prefers water (polar solvents), whereas the other prefers oil (non-polar solvents) (Tadros, 2014). Surfactants are used in variety of applications, both as part of food and edible components. Monostearate triglycerides are mostly employed in food and drug sector as viscosity modifiers and stabilizers that give food texture (Adewuyi *et al.*, 2014). Among the wide range on non-edible industrial applications of surfactants includes their role as stabilizing agents in mouth wash (Reshad *et al.*, 2009), aerosol (Modini *et al.*, 2013), corrosion inhibitor (Aslam *et al.*, 2018; Deyab, 2015; Shalabi *et al.*, 2019; Osman *et al.*, 2003), cosmetics and detergents (Bhalekar *et al.*, 2017; Rhein *et al.*, 2006).

Surfactants are important in drug delivery for a variety of reasons. Pharmaceutically approved solvents or surfactants are commonly used to improve the solubility of substances that are sparingly soluble in water. Surfactant-derived polymeric micelles have a number of unique properties that make them ideal drug carriers for a variety of medicines (Reddy *et al.*, 2013). The limited solubility of nearly half of the medications in biological fluids is still the principal barrier to oral, parenteral, and transdermal delivery. One of the most appealing possibilities for overcoming these difficulties is the incorporation of hydrophobic medicines into polymeric micelles consisting of surfactants (Mishra *et al.*, 2009).

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Quite a number of petroleum-based surfactants have been shown to be hazardous as well as increases the spread of other toxins in the environment to humans and the entire ecosystems (Emmanuel *et al.*, 2005, Metcalfe *et al.*, 2008). Despite their toxicity, some of these surfactants are still routinely used in a variety of ways. Some, such as linear alkyl benzene sulphonates (LAS) and alkyl phenol ethoxylates (APE), have been shown to persist in the environments after used (Badmus *et al.*, 2021). As a result of the toxicity and difficulties in degradation of these synthetic surfactants, a much more low-cost biosurfactants that are environmentally benign, and biodegradable and from renewable and widely available sources are still urgently needed (Tripathy and Mishra, 2016). Hence, this research is aimed at preparing biosurfactant from cotton seed oil as alternative to its synthetic counterparts.

2. Materials and Methods

2.1 Sample Preparation

A fully matured sample of cottonseed was collected sourced from Kwara State, Nigeria. The plant material was identified at the Herbarium of Plant Biology, University of Ilorin, Ilorin, Nigeria, where a specimen was deposited. The seeds were shelled to separate the seeds from the fibre, crushed, dried, and stored until they were needed for analysis.

2.2 Extraction and Physicochemical analysis of the Seed Oil

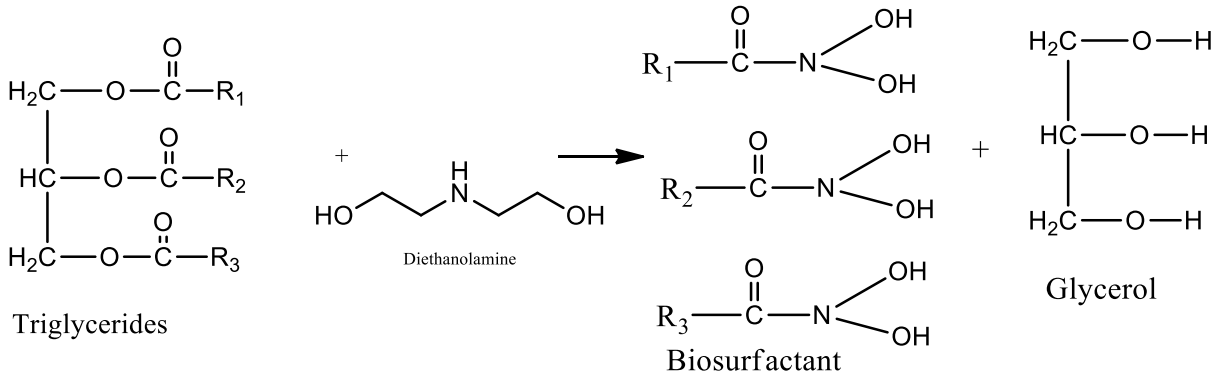
Oil extraction was carried out using n-hexane (1000 mL) according to the method of Zubair *et al.* (2018). 500g sample material of grounded dried seeds was extracted using Soxhlet extractor at 55°C for 7 hours. The oil was obtained using a rotary evaporator at 40°C. The Physicochemical properties such as acid value, iodine value, saponification value, peroxide value, free fatty acids were carried out using AOCS methods as described by Zubair *et al.*, (2018).

2.3 Preparation of Fatty Acid Methyl Ester (FAMEs)

The fatty acid methyl ester was prepared according to reported method of (Zubair *et al.*, 2018). 50 g oil was refluxed for one hour with 0.2 M methanolic HCl. At the end of the reflux, the FAMEs were obtained using n-hexane and concentrated using rotary evaporator.

2.4 Biosurfactant Preparation

This was carried out according to the method of Adewuyi *et al.* (2012). The reaction vessel was a round bottom Pyrex glass. The flask was equipped with a mechanical stirrer, thermometer and condenser. Esterified cotton seed oil and Diethanolamine was reacted at a molar ratio of 1:6 in the presence of sodium methoxide as a catalyst (2% by weight of diethanolamine and oil). The reaction was carried out at a temperature of 115 ^{0}C. At the end of the reaction, the mixture was allowed to cool and later dissolved in diethyl ether to recover the biosurfactant, separation was done using separating funnel. The ether phased was washed with water and passed over sodium sulphate. The resulting ether fraction was later concentrated using rotary evaporator. The scheme of the reaction is presented in Scheme 1.



Scheme 1: Preparation of the Biosurfactant

2.5 Spectroscopy Analysis

2.5.1 UV-Visible Spectroscopy Analysis of the Modified Substances

The seed oil, diethanolamine and prepared biosurfactants were analyzed using UV-Visible spectroscopy in dichloromethane respectively using uv-vis in the range of 300 nm up to 800 nm. A concentration of 1 µm was prepared in the solvents. The solvent was also used as the blank during the analysis procedure (Dhole *et al.*, 2015).

2.5.2 Fourier Transform Infrared (FT-IR) Spectroscopic Analysis

The seed oil, diethanolamine and biosurfactant were each subjected to infrared spectroscopic analyses separately in order to identify associated functional groups. The infrared spectra were recorded on Shimadzu 8400s according to the methods of Jain *et al.* (2016) using KBr pellet.

3. Result and Discussion

3.1 UV–visible spectroscopy data of the Biosurfactants

The preparation of the biosurfactant was established via UV–visible spectroscopy, with an increase or decrease in λmax between the reactants and final products taken into account. Table 2 and Figure 1 illustrate the results. The production of the SCOTS biosurfactant is confirmed by a bathochromic shift (red shift) in the SCOTS λmax (420 nm) due to π – π* and n – π* transitions in the λmax of cotton seed oil (318 nm) and 222 nm for diethanolamine. This was linked to the addition or combining of new auxochromes to extend the conjugation pair (Hikku *et al.*, 2018).

Table 1: UV-Visible Spectroscopic Characterization Data of the Biosurfactants

Samples	λmas (nm)
Cottonseed oil	318
Diethanolamine	222
Scots Biosurfactant	420

Scots – Cottonseed Biosurfactant

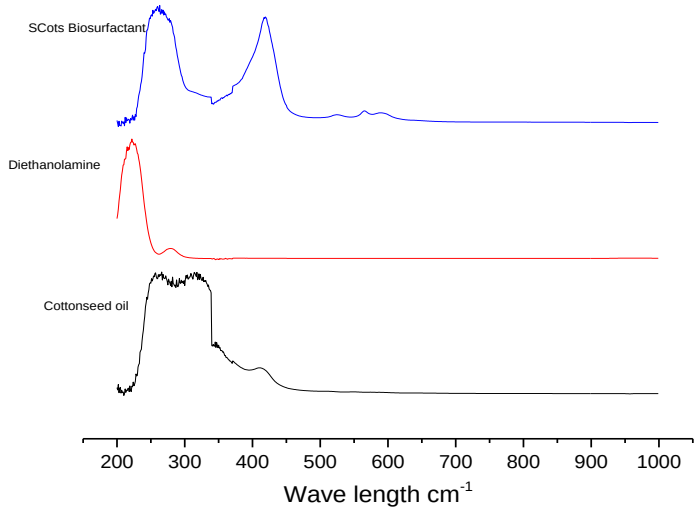


Figure 2: UV-Visible spectrum of SCOTS Biosurfactant

3.2 FT-IR Characterization Study of the Biosurfactants

Figure 3 presents the FT-IR spectra of cotton seed oil, SCOTS biosurfactants, and diethanolamine superimposed on one other. Table 3 shows Interpretation of FT-IR spectra. The biosurfactant preparation was confirmed by the results obtained and the emulsion stability test in Table 1. The less prominence of a hydroxyl group (O-H) peak in the seed oil, which was more apparent in diethanolamine and biosurfactants at 3394 cm^{-1} and 3404 cm^{-1}, respectively, suggested a functional group difference between the seed oils and the two other compounds (diethanolamine and biosurfactants). The absence of carbonyl group (C=O) in diethanolamine, which was found prevalent in the biosurfactants at 1620 cm^{-1}, also proved that biosurfactants were produced. This agrees with reported work of Adewuyi *et al.*, 2012 who characterized *Gliricida sepium* diethanolamide surfactant using FT-IR.

Table 2: FT-IR spectra Analysis of Cotton seed oil, Diethanolamine and Cotton seed biosurfactant

	Frequency of Absorption bands (cm^{-1})		
Functional groups	**Cotton seed oil**	**Diethanolamine**	**Cotton Biosurfactant**
C-O	1165	1064	1051
C-N		1301	1230 – 1300
C=O	1747		1620
N-H		1635	
C-H	2854 - 2926	2945	2926
O-H		3394	3385

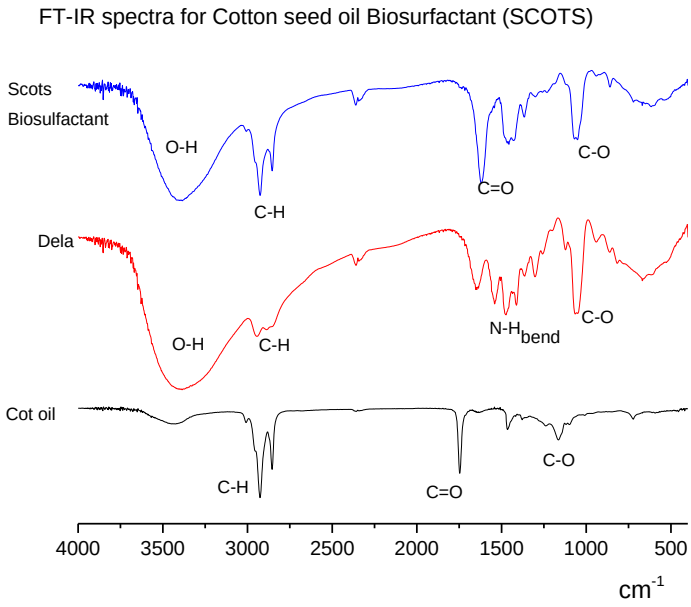


Figure 2: FT-IR Characterization of Sponge seed oil, Diethanolamine and Sponge seed oil Biosurfactant

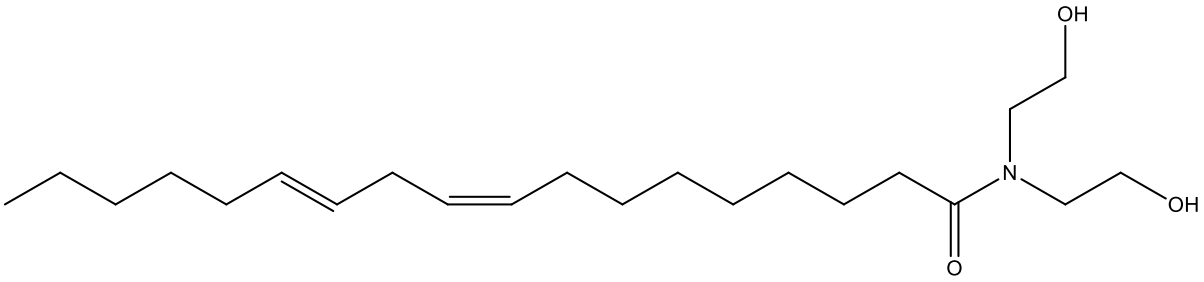


Figure 4: Proposed structures for SCOTS biosurfactant

Figure 3 present the proposed structure for SCOTS biosurfactant which was arrived at based on literature appraisals that confirmed linoleic acid as the most abundant fatty acids (Zubair *et al.*, 2021),

4. Conclusion

A large quantity of seeds available cheaply in Nigeria and many other tropical countries are often been discarded as waste, yet they offer solution to the problem of domestic-industrial competition for food and vegetable. The viability of the seeds oils to be processed into industrial grade products and replace dietary vegetable oils currently been used has been demonstrated by the preparation and characterization of the cotton seed oil biosurfactant. This implies the biosurfactants has the potentials to replace synthetic ones and helps against environmental pollution. Further work is ongoing to assess the toxicity and further characterization of the biosurfactant.

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